

**WELLS G&H SUPERFUND SITE  
WOBURN, MASSACHUSETTS**

**VAPOR INTRUSION ASSESSMENT WORK PLAN  
REVISION 1**

**AND**

**QUALITY ASSURANCE PROJECT PLAN FOR  
VAPOR INTRUSION ASSESSMENT**

**PREPARED FOR:**

**W.R. GRACE & CO. – CONN.  
62 WHITTEMORE AVENUE  
CAMBRIDGE, MASSACHUSETTS 02140**

**AND**

**UNIFIRST CORPORATION  
68 JONSPIN ROAD  
WILMINGTON, MA 01887**

**PREPARED BY:**

**GEOTRANS, INC.  
12 SPRING STREET  
SCHUYLERVILLE, NY 12871**

**GEOTRANS PROJECT NO. 117-3008059**

**MARCH 25, 2010**



**12 Spring Street, Suite 102, Schuylerville, New York 12871**

# VAPER INTRUSION ASSESSMENT WORK PLAN

## REVISION 1

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12 SPRING STREET  
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Superfund Records Center

SITE: Wells G&H 001

PREPARED: 8.5

OTHER: 479315



## TABLE OF CONTENTS

1	INTRODUCTION .....	1-1
1.1	BACKGROUND .....	1-2
1.1.1	HYDROGEOLOGIC SETTING .....	1-2
1.1.2	NATURE AND EXTENT OF GROUNDWATER CONTAMINATION.....	1-3
1.2	VAPOR INTRUSION ASSESSMENT ACTIVITIES .....	1-4
2	MONITORING WELL INSTALLATION AND GROUNDWATER SAMPLING .....	2-1
2.1	MONITORING WELL INSTALLATION AND SAMPLING .....	2-1
2.2	WELL INTEGRITY TESTING.....	2-2
2.3	GROUNDWATER SAMPLING .....	2-2
2.4	GROUNDWATER DATA TIER I VALIDATION.....	2-4
3	DATA EVALUATION.....	3-1
3.1	VIA REPORTING .....	3-1
4	SCHEDULE.....	4-1
5	REFERENCES .....	5-1

## **LIST OF TABLES**

<b>TABLE 2-1</b>	<b>NEW MONITORING WELLS PROPOSED FOR SAMPLING</b>
<b>TABLE 2-2</b>	<b>EXISTING MONITORING WELLS PROPOSED FOR SAMPLING</b>
<b>TABLE 2-3</b>	<b>PROPOSED VIA ANALYTES AND EPA-PROPOSED SCREENING CRITERIA</b>
<b>TABLE 2-4</b>	<b>SUMMARY OF SCREENED INTERVAL ELEVATIONS AND WATER-LEVEL ELEVATIONS FOR EXISTING WELLS PROPOSED FOR SAMPLING</b>

## **LIST OF FIGURES**

<b>FIGURE 1-1</b>	<b>SITE AREA MAP</b>
<b>FIGURE 1-2</b>	<b>2009 SHALLOW UNCONSOLIDATED DEPOSIT TETRACHLOROETHENE CONCENTRATION DISTRIBUTION</b>
<b>FIGURE 1-3</b>	<b>2009 SHALLOW UNCONSOLIDATED DEPOSIT TRICHLOROETHENE CONCENTRATION DISTRIBUTION</b>
<b>FIGURE 1-4</b>	<b>2009 SHALLOW UNCONSOLIDATED DEPOSIT 1,2-DICHLOROETHENE CONCENTRATION DISTRIBUTION</b>
<b>FIGURE 1-5</b>	<b>2009 SHALLOW UNCONSOLIDATED DEPOSIT VINYL CHLORIDE CONCENTRATION DISTRIBUTION</b>
<b>FIGURE 2-1</b>	<b>MONITORING WELLS FOR VI ASSESSMENT</b>

## 1 INTRODUCTION

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As requested by the United States Environmental Protection Agency (USEPA), this revised Vapor Intrusion Assessment (VIA) Work Plan is being submitted on behalf of W.R. Grace & Co. – Conn (Grace) and UniFirst Corporation (UniFirst) to assess the potential for vapor intrusion from volatilization of compounds in groundwater in a portion of the Northeast Quadrant of the Wells G&H Superfund Site (Site). The Site, Grace and UniFirst properties, and the VIA study area are shown on Figure 1-1.

As USEPA stated in its 2004 Five-Year Review (USEPA, 2004), the potential vapor intrusion pathway in this portion of the Site has been evaluated repeatedly in the past 20 years. The area was the subject of indoor air sampling in July 1989 and October 1991, followed by an evaluation of those data in 1995 (ATSDR, 1995). In 1989, 1991, and 1995, USEPA and ATSDR concluded that the vapor intrusion pathway presented no apparent public health hazard. USEPA reexamined the historical data in 2004 and reached the same conclusion based on current toxicological information. In 2004, USEPA also conducted vapor intrusion modeling using then current groundwater contaminant concentrations, measured as part of the long-term monitoring programs instituted by Grace and UniFirst in the Northeast Quadrant. USEPA concluded in 2004 that estimated risks based on its modeling results “are within or below EPA risk management guidelines, confirming earlier results based on indoor air sampling” (USEPA, 2004).

The potential for vapor intrusion in the study area will be re-evaluated through an initial groundwater sampling event at specific locations, followed by a second round of sampling approximately six months after the initial sampling round. A report describing the results of the assessment will be provided to USEPA within 60 days after receipt of the validated analytical data from the second groundwater sampling event. To the extent possible, one of these two VIA sampling events will be scheduled to coincide with the annual sampling of monitoring wells located on the Grace and UniFirst properties and in the Northeast Quadrant of the Wells G&H Site.

## **1.1 BACKGROUND**

### **1.1.1 HYDROGEOLOGIC SETTING**

The Northeast Quadrant of the Site is underlain by glacially derived unconsolidated deposits and crystalline bedrock. The unconsolidated deposits beneath the eastern portion of the Northeast Quadrant vary in thickness and composition, but generally consist of ablation and lodgment till comprised of low-permeability over-consolidated poorly-sorted sand, gravel, silt, and clay. In the central portion of the Aberjona River valley more permeable stratified sand and gravel generally overlie the till deposits. Fractures in the crystalline bedrock allow relatively high well yields, such as that achieved by UniFirst recovery well UC22, in some areas of the Site.

Prior to implementation of the combined groundwater remedies, groundwater from the Grace and UniFirst properties flowed westerly and southwesterly beneath the study area towards the Aberjona River.

Grace and UniFirst began operating coordinated groundwater remedies in the Northeast Quadrant of the Site on September 30, 1992. The UniFirst on-property groundwater recovery and treatment system has sustained a widespread capture zone that extends well beyond the UniFirst property boundaries in unconsolidated deposits and in bedrock. The system is preventing contaminated groundwater from flowing off the UniFirst property and reducing volatile organic compound (VOC) concentrations in groundwater in unconsolidated deposits on the UniFirst property. In addition the deep bedrock extraction well on the UniFirst property (UC22) is recovering contaminated groundwater throughout the zone of capture in the Northeast Quadrant, including from deep bedrock beneath the Grace property.

The Grace on-property groundwater recovery and treatment system has prevented contaminated groundwater from flowing off the Grace property in the unconsolidated deposits and shallow bedrock, and significantly reduced VOC concentrations in groundwater in these deposits across the property. Contaminated groundwater in the deep bedrock beneath the Grace property is captured by UC22, the deep bedrock recovery well located on the nearby UniFirst property.

### 1.1.2 NATURE AND EXTENT OF GROUNDWATER CONTAMINATION

The combined Grace and UniFirst groundwater remedies affect a large portion of the Wells G&H Site and have significantly reduced the contaminant concentrations in groundwater in the Northeast Quadrant of the Site. These effects are apparent from the results of extensive water-level measurements and analysis of groundwater quality samples collected from monitoring and recovery wells, and from treatment system influent and effluent water quality samples collected since the recovery wells began operating in September 1992 (GeoTrans, 2008; Harvard Project Services, 2008).

The Northeast Quadrant groundwater remedy performance monitoring data indicate that VOC concentrations in the saturated unconsolidated deposits in the study area have been substantially reduced since the recovery wells began operation. Along the Grace property western boundary, VOC concentrations at all unconsolidated deposit monitoring wells and all recovery wells have been reduced to less than the cleanup goals. Along the Grace southern property boundary, VOC concentrations have been substantially reduced and are at or approaching the cleanup goals in all recovery wells. VOC concentrations at monitoring well G24S remain above the cleanup goals. Along the UniFirst western property boundary, VOC concentrations in all unconsolidated deposit water table monitoring wells are below the cleanup goals. Along the UniFirst southern property boundary, the VOC concentrations at monitoring well S71S have been reduced substantially, but remain above cleanup goals. Long-term monitoring data indicate that VOC concentrations at the unconsolidated deposit water table wells at S63 and GO1, located south of the Dewey Avenue neighborhood, were reduced to less than the laboratory detection limits by 1993 and 1994, respectively. In the two unconsolidated deposit wells located directly west of the Dewey Avenue neighborhood, S81S and S82, with the exception of tetrachloroethene (PCE) at S81S, VOC concentrations have been reduced to less than the cleanup goals. Figures 1-2 through 1-5 show the current distribution of PCE, trichloroethene (TCE), cis-1,2-dichloroethene (DCE), and vinyl chloride (VC) concentrations in unconsolidated deposits groundwater in the VIA study area. These figures were prepared using the 2009 monitoring data augmented with data from the most recent samples collected from monitoring wells GO1S, S63S, and S82. Long-term monitoring sampling of wells GO1S and S63S was stopped in 1996 following several successive samples with concentrations below the ROD cleanup levels. USEPA collected samples from S63S and S82 in 2002.

## **1.2 VAPOR INTRUSION ASSESSMENT ACTIVITIES**

The VIA will include the installation and sampling of new shallow groundwater monitoring wells in addition to sampling existing shallow monitoring wells to evaluate the potential risks of vapor intrusion in the study area. Groundwater samples will be analyzed for VOCs by USEPA Method 8260B, with selected ion monitoring mode (SIM) where necessary and possible to achieve applicable reporting limits.

Grace and UniFirst have designed these investigations to address the USEPA request related to evaluating the potential for vapor intrusion in the study area by determining if VOCs are present in the shallow groundwater at sufficient concentrations to pose a vapor intrusion risk to nearby structures. To collect the information to assess the potential for vapor intrusion, the following work is proposed:

- Install two shallow monitoring wells west of the UniFirst property;
- Install nine shallow monitoring wells in the Dewey Avenue neighborhood;
- Install two shallow monitoring wells south of the Grace property; and
- Collect groundwater samples for VOC analyses from all newly installed wells and from seven existing water table groundwater monitoring wells.

In addition, UniFirst intends to collect shallow groundwater data concurrently with the VIA to evaluate shallow groundwater VOC concentrations at the water table using existing, useable water-table monitoring wells on the UniFirst property (The Johnson Company, 2010).

Section 2 of this Work Plan describes the work related to the installation and sampling of the monitoring wells and Section 3 describes the vapor intrusion assessment to be done. Section 4 presents a proposed schedule, and Section 5 provides references cited in this Work Plan.

## **2 MONITORING WELL INSTALLATION AND GROUNDWATER SAMPLING**

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Grace and UniFirst propose to install 13 shallow groundwater monitoring wells and collect groundwater samples from them. In addition, groundwater samples will be collected from seven existing groundwater monitoring wells in the study area to further assess the potential for vapor intrusion in the VIA study area. These data will reflect the VOC concentrations in the groundwater at the water table within the VIA study area.

### **2.1 MONITORING WELL INSTALLATION AND SAMPLING**

To assess the potential for vapor intrusion in the study area, 13 2-inch diameter shallow monitoring wells will be installed. The proposed well locations are shown on Figure 2-1. The wells will be drilled to a depth of approximately ten feet below the water table. It is anticipated that monitoring well depths will be 15 to 30 feet below ground surface. It is also anticipated that most, if not all, of the wells will be screened in the unconsolidated deposits. At locations where the water table is near or below the bottom of the unconsolidated deposits, the wells will extend into bedrock. Drilling will be conducted using roto sonic and/or rotary techniques. Two-inch diameter polyvinyl chloride (PVC) wells with ten feet of ten-slot screen will be installed such that the water table is within the screened interval of the well. The wells will be completed to grade with flush-mount well caps. Well development will take place at the time of installation. All development water will be contained and treated at the Grace or UniFirst groundwater treatment system. Detailed procedures for well installation and development are presented in Appendix 2 of the Quality Assurance Project Plan for Vapor Intrusion Assessment (QAPP) (GeoTrans, 2010). Well installation details will be recorded in field books and presented on Field Boring Logs.

Headspace field screening will be conducted during drilling with a photo-ionization detector (PID). Drill cuttings will be screened at five-foot intervals. Detailed procedures for dealing with drill cuttings are presented in Appendix 2 of the QAPP. Unconsolidated deposit samples and bedrock cores will be described by GeoTrans field personnel.

All proposed monitoring wells within the Dewey Avenue neighborhood will be installed on City of Woburn property. The proposed wells south of the Grace property and west of the UniFirst property will be installed on Cummings property. Appropriate local permits, access

agreements, and police detail, if required, will be arranged prior to initiating work conducted in or near public roadways or on public property.

## **2.2 WELL INTEGRITY TESTING**

Prior to collecting samples from existing monitoring wells not included in the Grace and UniFirst Northeast Quadrant remedy long-term monitoring programs (GO1S, K55S, K60S, S21, S22, and S63S), well integrity tests will be conducted. These tests will include a visual inspection of the well, depth measurement to confirm well depth, and a slug test with water level measurements to confirm the hydraulic connection of the well to the saturated unconsolidated deposits. If the well integrity tests indicate that well development is necessary, the wells will be redeveloped by bailing. These wells will not be sampled if the well integrity tests indicate that the wells are not usable. Detailed procedures for well integrity testing are presented in Appendix 2 of the QAPP (GeoTrans, 2010)

## **2.3 GROUNDWATER SAMPLING**

Two rounds of groundwater samples will be collected from the new monitoring wells listed in Table 2-1, and the seven existing monitoring wells listed in Table 2-2. The wells proposed for sampling are shown on Figure 2-1. In order to collect samples that are representative of the groundwater quality at the water table, the sampling will be performed using the low-flow groundwater sampling methodology described in Appendix 2 of the QAPP (GeoTrans, 2010). Groundwater sampling will begin approximately two weeks after well development is completed. The groundwater samples will be sent to a laboratory for analysis of the VOC analytes listed in Table 2-3. The samples will be analyzed using USEPA Method 8260B, with SIM where necessary and possible to achieve applicable reporting limits. Table 2-3 represents all VOCs where the maximum concentration detected in groundwater in monitoring wells located on either the Grace or UniFirst properties was greater than the EPA-proposed Screening Criteria (USEPA, 2010) or where the previous detection limits were greater than the EPA-proposed Screening Criteria. These groundwater data will provide shallow groundwater quality to aid in the VIA.

Analytical methods to be used for the groundwater samples, sample bottles, preservatives, and laboratory holding times are specified in the QAPP (GeoTrans, 2010). All



data will be recorded in field log books and presented on the appropriate field forms as found in the QAPP (GeoTrans, 2010). Chain-of-custody and sample packaging and shipment procedures that will be followed are outlined in the QAPP (GeoTrans, 2010).

**Table 2-1 New Monitoring Wells Proposed for Sampling**

UG8	UG12	UG16	UG20
UG9	UG13	UG17	
UG10	UG14	UG18	
UG11	UG15	UG19	

**Table 2-2 Existing Monitoring Wells Proposed for Sampling**

GO1S	K60S	S22	S81S
K56S	S21	S63S	

**Table 2-3 Proposed VIA Analytes and EPA-Proposed Screening Criteria**

Compound	Screening Criteria (µg/L)	Compound	Screening Criteria (µg/L)
1,1,1-Trichloroethane	714	Chlorobenzene	33.3
1,1,2-Trichloroethane	4.11	Chloroform	0.705
1,1-Dichloroethane	6.61	Cis-1,2-dichloroethene	no value (0.5)
1,1-Dichloroethene	18.7	Ethylbenzene	3.04
1,2,4-Trimethylbenzene	2.8	Isopropylbenzene	0.844
1,2-Dibromoethane	0.135	Methylene chloride	57.5
1,2-Dichloroethane	2.34	Naphthalene	3.98
1,2-Dichloropropane	2.12	Tetrachloroethene	0.55
1,3-Dichlorobenzene	no value (0.5)	Toluene	1850
1,4-Dichlorobenzene	2.25	Trans-1,2-dichloroethene	15.8
Benzene	1.36	Trans-1,3-Dichloropropene	0.84
Bromodichloromethane	0.94	Trichloroethene	2.89
Bromoform	101	Vinyl chloride	0.145
Carbon tetrachloride	0.135	Xylenes (total)	35.7

The existing monitoring wells proposed for sampling are located adjacent to the Dewey Avenue neighborhood. The wells are all screened across or near the water table. Table 2-4 summarizes the elevations of the screened intervals and recent water level elevations. Water quality data collected for this VIA will be supplemented with data from wells located along the southern and western UniFirst property boundaries collected as part of the UniFirst property vapor intrusion evaluation.

**Table 2-4 Summary of Screened Interval Elevations and Water-Level Elevations for Existing Wells Proposed for Sampling**

Well	Top of Screen Elevation (Ft.)	Bottom of Screen Elevation (Ft.)	Water Level Elevation (Ft.)	Water Level Measurement Date
GO1S	65.2	55.2	59.52	4/13/2009
K55S	67.7	66.7	NA	NA
K60S	58.1	57.1	NA	NA
S21	73.7	46.2	60.2 – 57.1	1991 Pilot Test
S22	81	41	75.4-65.6	1991 Pilot test
S63S	58	48	58.66	4/13/2009
S81S	44.7	34.7	52.45	4/14/2009
NA - Not Available				

## 2.4 GROUNDWATER DATA TIER I VALIDATION

To assure groundwater data meet all data quality objectives and are representative of Site conditions, a Region 1 Tier III data validation will be conducted. Data validation procedures are described in detail in Form R of the QAPP (GeoTrans, 2010). The data will be checked and assessed for completeness, accuracy, and usability. The quality control sampling results will be reviewed using both statistical and qualitative evaluations. Data will be qualified, as necessary, based on the criteria summarized in the QAPP (GeoTrans, 2010).

## 2.5 HEALTH & SAFETY PLAN

The Health and Safety Plan for the Northeast Quadrant of the Site (HASP) (GeoTrans, 2004) provides general health and safety information and establishes the minimum health and

safety related procedures and requirements associated with implementing this VIA. The HASP establishes the minimum health and safety related information for personnel engaged in activities at the Site. At the discretion of each firm/organization engaged in Site activities, the information presented may be supplemented by addenda or contractor-specific health and safety plans addressing the task(s) to be performed by their respective personnel.

## **2.6 COMMUNITY RELATIONS**

Because off-property work will be conducted as part of this VIA scope, it is expected that USEPA will provide public notifications prior to commencement of the work. At Superfund sites, USEPA has the primary responsibility for taking the lead role in community relations. The Grace and UniFirst team will actively support USEPA with community relations.

### **3 DATA EVALUATON**

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The groundwater-quality data collected will be compared with the EPA-proposed Screening Criteria as shown in Table 2-3. If the water-quality data indicate that VOC concentrations are below the EPA-proposed Screening Criteria identified in Table 2-3, no further vapor intrusion assessment work will be conducted in the study area. If the water quality data indicate that there is the potential for vapor intrusion into residential or commercial buildings in the study area, Grace and UniFirst will propose a phased approach and strategy to continue with further soil gas surveys in the study area. Additional investigations may include building surveys, sub-slab soil gas collection, or mathematical modeling.

#### **3.1 VIA REPORTING**

The results of the VIA will be presented in the VIA Report. The report will include:

- Objectives of the VIA;
- An overview of the monitoring well installation;
- Details regarding the groundwater quality samples and results; and
- Conclusions and recommendations.

## **4 SCHEDULE**

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Grace and UniFirst propose to install the new monitoring wells in the spring of 2010. The installation of the monitoring wells is anticipated to take approximately two weeks to complete. The first sampling event will be conducted two weeks after the development of the new wells is completed, and the second sampling event is proposed to be performed six months later, in the fall of 2010. UniFirst is submitting separately a work plan to meet USEPA's request for an assessment of the potential vapor intrusion pathway on the UniFirst property (The Johnson Company, 2010). Grace and UniFirst intend to coordinate scheduling of field work so that shallow groundwater sampling can be conducted concurrently under both work plans, to the extent possible. A report detailing the VIA will be provided to USEPA within 60 days after receipt of the validated analytical data from the second groundwater sampling event.

## 5 REFERENCES

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- ATSDR, 1995, Public Health Assessment Addendum, Wells G&H, Woburn, Middlesex County, Massachusetts. U.S. Department of Public Health and Human Service, Public Health Service Agency for Toxic Substance and Disease Registry. December 1995.
- Harvard Project Services LLC, 2008. RD/RA Year 16 Annual Report for the UniFirst Site. November 12, 2008.
- GeoTrans, 2004, Health and Safety Plan Update For Northeast Quadrant of Wells G&H Site Long Term Monitoring Plan, April 21, 2004
- GeoTrans, 2008, W.R. Grace Remedial Action Annual Report, November 14, 2008.
- GeoTrans, 2010, Quality Assurance Project Plan, Vapor Intrusion Assessment, March 25, 2010.
- US Environmental Protection Agency, 2004, Second Five Year Review Report for Wells G&H Superfund Site, Woburn, Middlesex County, Massachusetts, September 30, 2004 (USEPA, 2004)
- Public Health Service Agency for Toxic Substances and Disease Registry, 1995, Public Health Assessment Addendum, Wells G&H, Woburn, Middlesex County,, Massachusetts, December 1995 (ATSDR, 1995).
- The Johnson Company, Inc., 2010. Indoor Air Quality and Vapor Intrusion Assessment Scope of Work, Revision 2, UniFirst Source Area Property, Wells G&H Superfund Site, Woburn, Massachusetts, March 2010.
- US Environmental Protection Agency, 2010, Letter re: EPA review of UniFirst's draft VI Scope of Work, dated October 9, 2009. February 25, 2010.
- US Environmental Protection Agency, 2009, Response to Source Area Properties Annual Reports, May 14, 2009.
- US Environmental Protection Agency, 2004, User's Guide for Evaluating Subsurface Vapor Intrusion Into Buildings (USEPA, 2004)
- US Environmental Protection Agency, 2002, Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (USEPA, 2002)







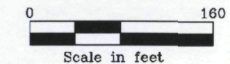
NOTE: PROPERTY BOUNDARY INFORMATION IN THE DEWEY AVE. NEIGHBORHOOD FROM MAP TITLED "TAX MAP 26 OF THE CITY OF WOBURN MASSACHUSETTS" PREPARED BY THE CITY OF WOBURN ENGINEERING DEPARTMENT




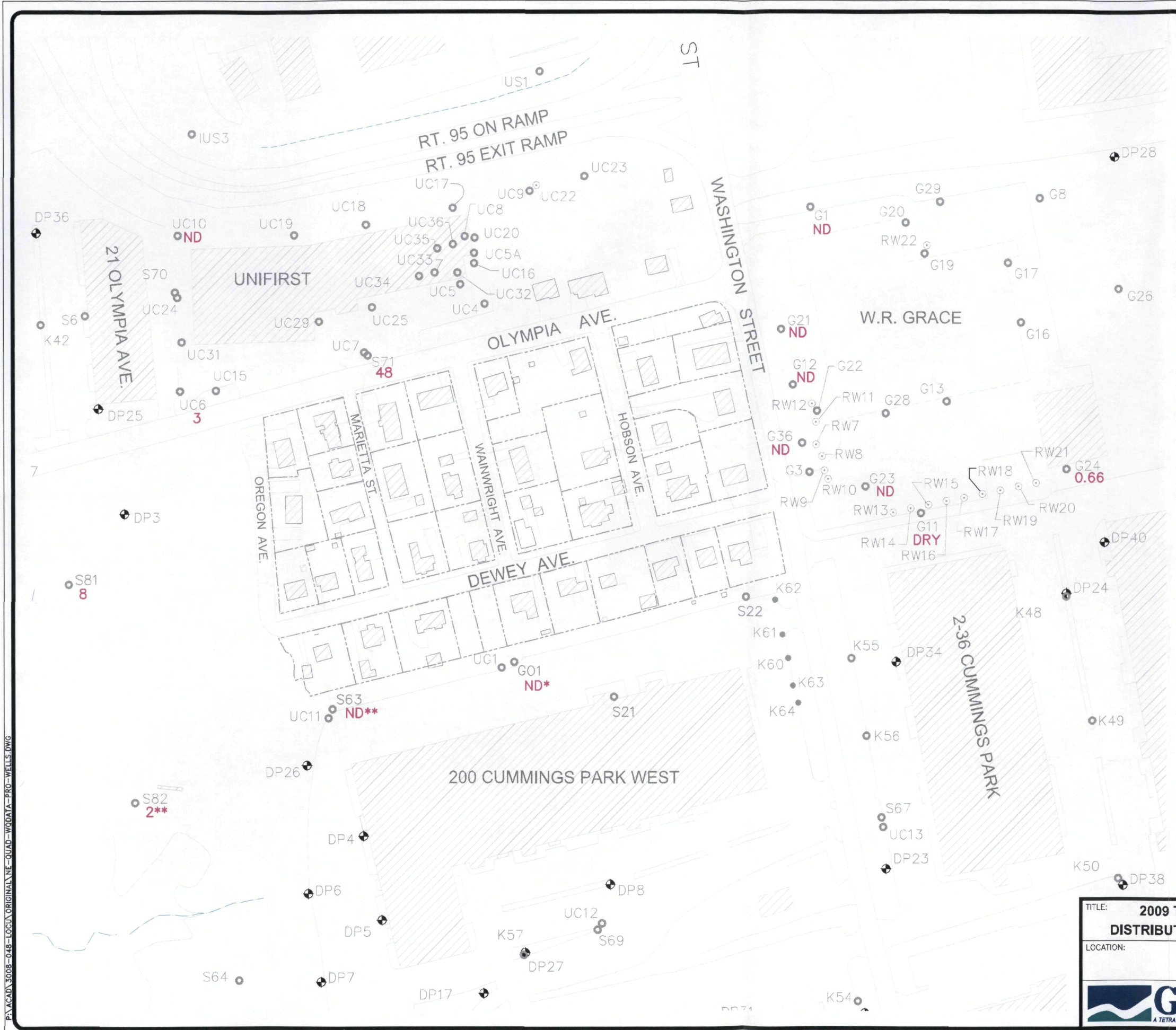
### EXPLANATION

- APPROXIMATE PROPERTY BOUNDARY DEWEY AVE. NEIGHBORHOOD
- RW10 ● RECOVERY WELL LOCATION
- UC6 ● MONITORING WELL LOCATION
- 0.63 TETRACHLOROETHENE (ug/L)
- 10/9.8-DUPLICATE SAMPLE
- ND NOT DETECTED AT DETECTION LIMIT
- J ESTIMATED VALUE
- \* G01 LAST SAMPLED 5/7/1996
- \*\* S63S & S82 LAST SAMPLED 10/15/2002

CONCENTRATIONS PRESENTED ARE FROM THE SHALLOWEST UNCONSOLIDATED DEPOSIT WELL AT EACH WELL CLUSTER



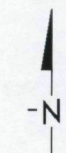
TITLE: 2009 TETRACHLOROETHENE CONCENTRATIONS DISTRIBUTED IN SHALLOW UNCONSOLIDATED DEPOSITS			
LOCATION: Wells G & H, Woburn, MA.			
	APPROVED	JRB	FIGURE <b>1-2</b>
	DRAFTED	RMK	
	PROJECT#	117-3008048	
	DATE	OCTOBER 2009	



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NOTE: PROPERTY BOUNDARY INFORMATION IN THE DEWEY AVE. NEIGHBORHOOD FROM MAP TITLED "TAX MAP 26 OF THE CITY OF WOBURN MASSACHUSETTS" PREPARED BY THE CITY OF WOBURN ENGINEERING DEPARTMENT

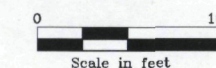



# EXPLANATION

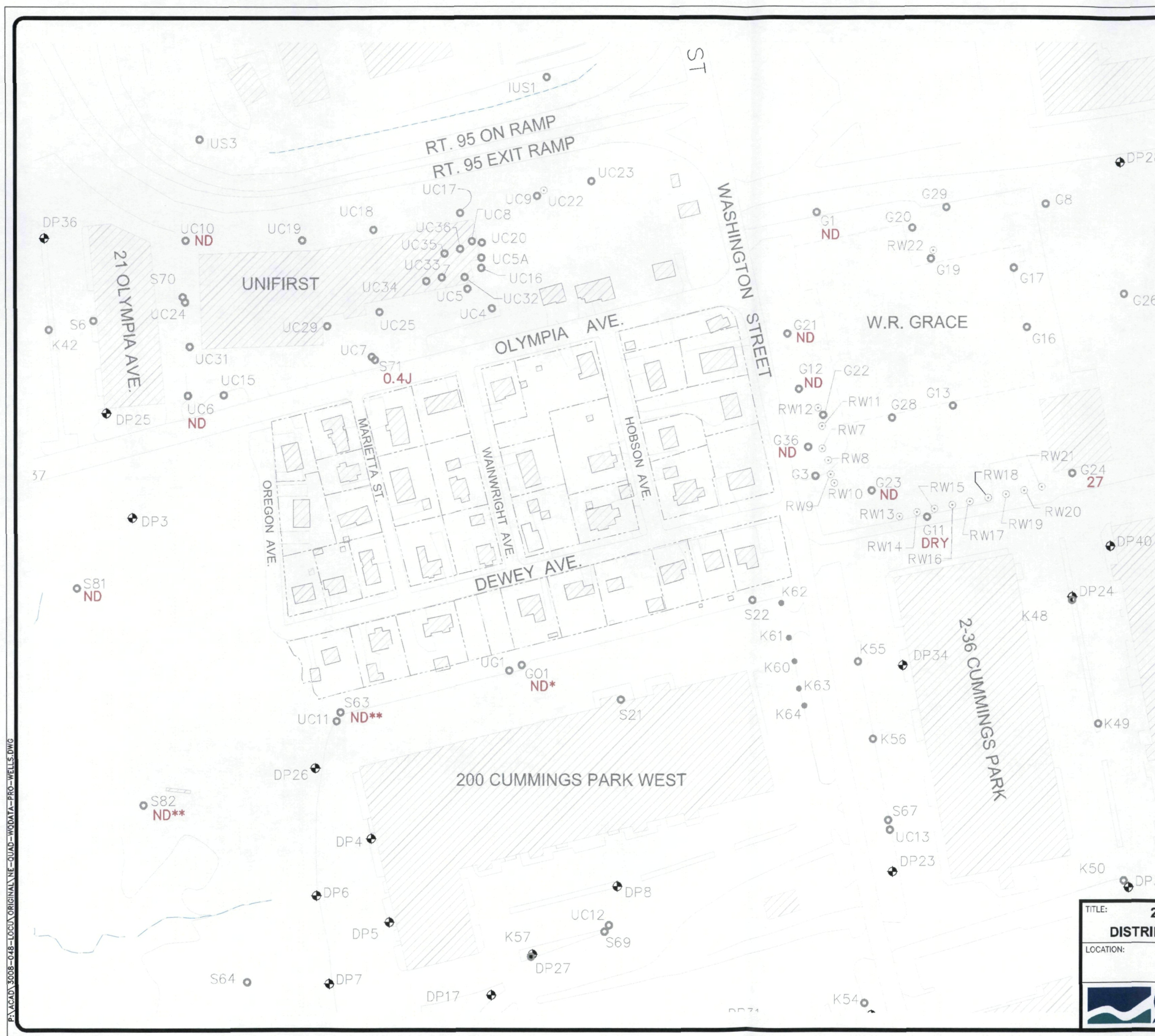
- APPROXIMATE PROPERTY BOUNDARY DEWEY AVE. NEIGHBORHOOD
- RW10⊙ RECOVERY WELL LOCATION
- UC6⊙ MONITORING WELL LOCATION
- 5.4 TRICHLOROETHENE (ug/L)
- 3.1/3.2 -DUPLICATE SAMPLE
- ND NOT DETECTED AT DETECTION LIMIT
- J ESTIMATED VALUE

\* G01 LAST SAMPLED 5/7/1996  
 \*\* S63S & S82 LAST SAMPLED 10/15/2002

CONCENTRATIONS PRESENTED ARE FROM THE SHALLOWEST UNCONSOLIDATED DEPOSIT WELL AT EACH WELL CLUSTER



TITLE: 2009 TRICHLOROETHENE CONCENTRATIONS DISTRIBUTED IN SHALLOW UNCONSOLIDATED DEPOSITS			
LOCATION: Wells G & H, Woburn, MA.			
	APPROVED	JRB	FIGURE <b>1-3</b>
	DRAFTED	RMK	
	PROJECT#	117-3008048	
	DATE	OCTOBER 2009	



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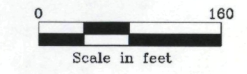
NOTE: PROPERTY BOUNDARY INFORMATION IN THE DEWEY AVE. NEIGHBORHOOD FROM MAP TITLED "TAX MAP 26 OF THE CITY OF WOBURN MASSACHUSETTS" PREPARED BY THE CITY OF WOBURN ENGINEERING DEPARTMENT




# EXPLANATION

- APPROXIMATE PROPERTY BOUNDARY DEWEY AVE. NEIGHBORHOOD
- RW10⊙ RECOVERY WELL LOCATION
- UC6⊙ MONITORING WELL LOCATION
- 27 1,2-DICHLOROETHENE (TOTAL) (ug/L)
- 1.5/1.7-DUPLICATE SAMPLE
- ND NOT DETECTED AT DETECTION LIMIT
- J ESTIMATED VALUE
- \* G01 LAST SAMPLED 5/7/1996
- \*\* S63S & S82 LAST SAMPLED 10/15/2002

CONCENTRATIONS PRESENTED ARE FROM THE SHALLOWEST UNCONSOLIDATED DEPOSIT WELL AT EACH WELL CLUSTER



TITLE: 2009 1,2-DICHLOROETHENE CONCENTRATIONS DISTRIBUTED IN SHALLOW UNCONSOLIDATED DEPOSITS			
LOCATION: Wells G & H, Woburn, MA.			
	APPROVED	JRB	FIGURE <b>1-4</b>
	DRAFTED	RMK	
	PROJECT#	117-3008048	
	DATE	OCTOBER 2009	



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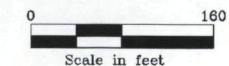
NOTE: PROPERTY BOUNDARY INFORMATION IN THE DEWEY AVE. NEIGHBORHOOD FROM MAP TITLED "TAX MAP 26 OF THE CITY OF WOBURN MASSACHUSETTS" PREPARED BY THE CITY OF WOBURN ENGINEERING DEPARTMENT




# EXPLANATION

- APPROXIMATE PROPERTY BOUNDARY DEWEY AVE. NEIGHBORHOOD
- RW10⊙ RECOVERY WELL LOCATION
- UC6⊙ MONITORING WELL LOCATION
- ND VINYL CHLORIDE (ug/L)
- ND/ND -DUPLICATE SAMPLE
- ND NOT DETECTED AT DETECTION LIMIT
- J ESTIMATED VALUE
- \* G01 LAST SAMPLED 5/7/1996
- \*\* S63S & S82 LAST SAMPLED 10/15/2002

CONCENTRATIONS PRESENTED ARE FROM THE SHALLOWEST UNCONSOLIDATED DEPOSIT WELL AT EACH WELL CLUSTER



TITLE: 2009 VINYL CHLORIDE CONCENTRATIONS DISTRIBUTED IN SHALLOW UNCONSOLIDATED DEPOSITS			
LOCATION: Wells G & H, Woburn, MA.			
		APPROVED	JRB
		DRAFTED	RMK
		PROJECT#	117-3008048
		DATE	OCTOBER 2009
			FIGURE <b>1-5</b>

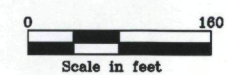


NOTE: PROPERTY BOUNDARY INFORMATION IN THE DEWEY AVE. NEIGHBORHOOD FROM MAP TITLED "TAX MAP 26 OF THE CITY OF WOBURN MASSACHUSETTS" PREPARED BY THE CITY OF WOBURN ENGINEERING DEPARTMENT

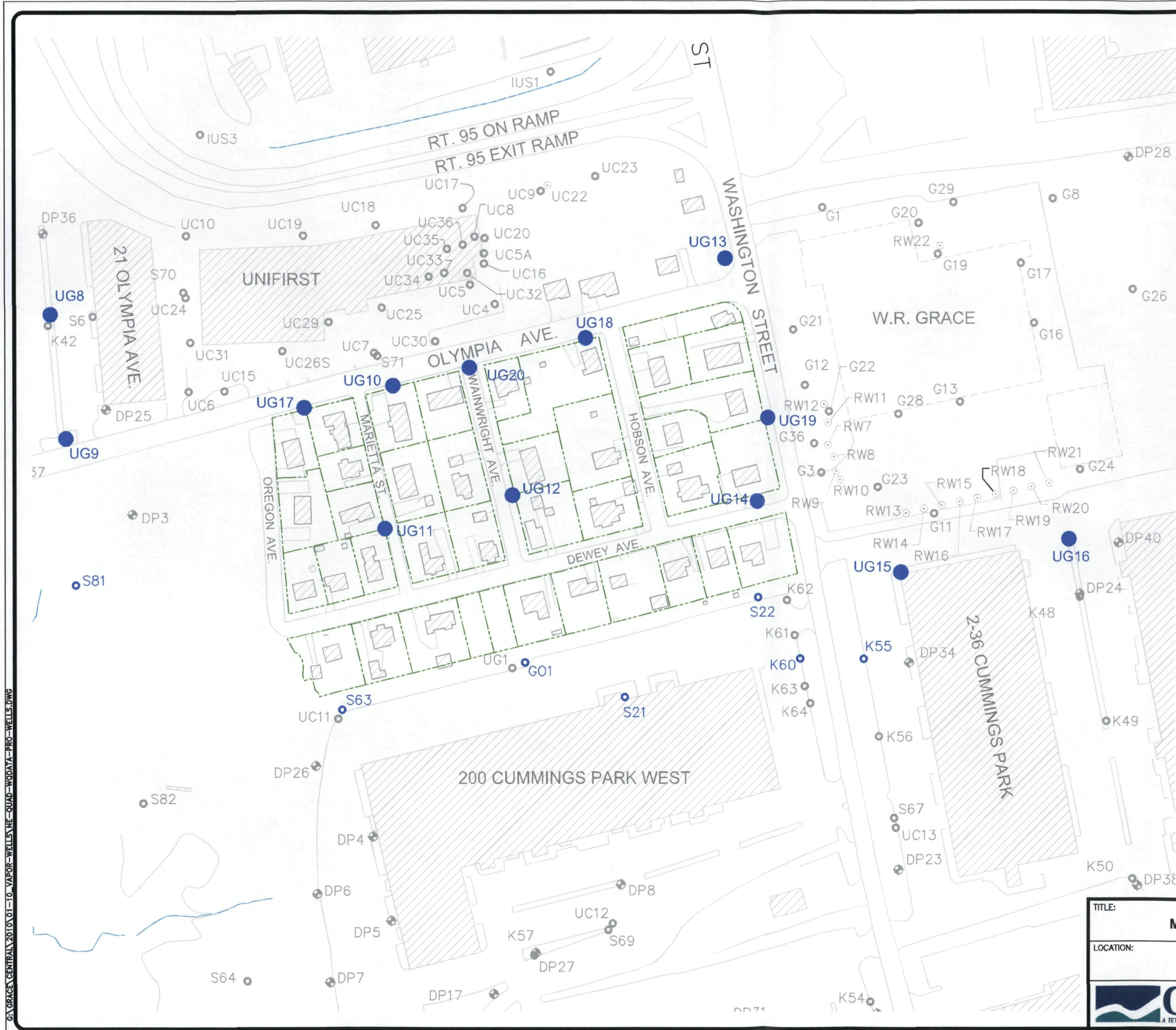


### EXPLANATION

- APPROXIMATE PROPERTY BOUNDARIES DEWEY AVE. NEIGHBORHOOD
- RW10 RECOVERY WELL LOCATION
- UC6 MONITORING WELL LOCATION
- UG14 PROPOSED NEW WATER TABLE MONITORING WELL
- S81 SHALLOWEST WELL AT EXISTING LOCATION PROPOSED FOR SAMPLING



<b>TITLE:</b> <b>MONITORING WELLS FOR VI ASSESSMENT</b>											
<b>LOCATION:</b> <b>Wells G &amp; H, Woburn, MA.</b>											
<b>GeoTrans, Inc.</b> <small>A TETRA TECH COMPANY</small>		<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="padding: 2px;">APPROVED</td> <td style="padding: 2px;">JRB</td> </tr> <tr> <td style="padding: 2px;">DRAFTED</td> <td style="padding: 2px;">RMK</td> </tr> <tr> <td style="padding: 2px;">PROJECT#</td> <td style="padding: 2px;">117-3008048</td> </tr> <tr> <td style="padding: 2px;">DATE</td> <td style="padding: 2px;">FEB 2010</td> </tr> </table>	APPROVED	JRB	DRAFTED	RMK	PROJECT#	117-3008048	DATE	FEB 2010	<b>FIGURE</b> <b>2-1</b>
APPROVED	JRB										
DRAFTED	RMK										
PROJECT#	117-3008048										
DATE	FEB 2010										



G:\GRACE CENTRAL\2010\01-10-VAPOR-WELLS\NE-QUAD-WO\DATA-PRO-WELLS.DWG



QUALITY ASSURANCE  
PROJECT PLAN FOR VI  
ASSESSMENT

**WELLS G&H SUPERFUND SITE  
WOBURN, MASSACHUSETTS**

**QUALITY ASSURANCE PROJECT PLAN FOR  
VAPOR INTRUSION ASSESSMENT**

PREPARED FOR:

W.R. GRACE & CO. – CONN.  
62 WHITTEMORE AVENUE  
CAMBRIDGE, MASSACHUSETTS 02140

AND

UNIFIRST CORPORATION  
68 JONSPIN ROAD  
WILMINGTON, MA 01887

PREPARED BY:

GEOTRANS, INC.  
12 SPRING STREET  
SCHUYLERVILLE, NY 12871

GEOTRANS PROJECT NO. 117-3008059

MARCH 25, 2010



12 Spring Street, Suite 102, Schuylerville, New York 12871

**QUALITY ASSURANCE PROJECT PLAN FOR**  
**VAPOR INTRUSION ASSESSMENT**

---

WELLS G&H SUPERFUND SITE  
WOBURN, MASSACHUSETTS

PREPARED FOR:

W.R. GRACE & CO. – CONN.  
62 WHITTEMORE AVENUE  
CAMBRIDGE, MASSACHUSETTS 02140

AND

UNI-FIRST CORPORATION  
68 JONSPIN ROAD  
WILMINGTON, MA 01887

PREPARED BY:

GEOTRANS, INC.  
12 SPRING STREET  
SCHUYLERVILLE, NY 12871

## TABLE OF CONTENTS

FORM A: TITLE AND APPROVAL PAGE AND INTRODUCTION .....	1
FORM B: PROJECT ORGANIZATION AND RESPONSIBILITY .....	4
FORM C: PROBLEM DEFINITION .....	5
FORM D: PROJECT DESCRIPTION/TIMELINE .....	7
FORM E: SAMPLING DESIGN.....	9
FORM F: SAMPLING AND ANALYTICAL METHODS REQUIREMENTS.....	11
FORM G: METHOD AND SOP REFERENCE TABLE .....	12
FORM H: FIELD EQUIPMENT CALIBRATION AND CORRECTIVE ACTION.....	13
FORM I: LABORATORY EQUIPMENT CALIBRATION AND CORRECTIVE ACTION ..	16
FORM J: SAMPLE HANDLING AND CUSTODY REQUIREMENTS .....	17
FORM K: ANALYTICAL SENSITIVITY AND PROJECT CRITERIA .....	22
FORM L: FIELD QUALITY CONTROL.....	24
FORM M: LABORATORY QUALITY CONTROL.....	25
FORM N: DATA MANAGEMENT AND DOCUMENTATION .....	27
FORM O: ASSESSMENT AND RESPONSE ACTIONS.....	30
FORM P: PROJECT REPORTS .....	32
FORM Q: FIELD DATA EVALUATION.....	33
FORM R: LABORATORY DATA EVALUATION .....	34
FORM S: DATA USABILITY AND PROJECT EVALUATION .....	37
REFERENCES.....	39

## LIST OF FIGURES

FIGURE 1      MONITORING WELLS FOR VI ASSESSMENT

## LIST OF APPENDICES

APPENDIX 1    ANALYTICAL METHOD SOPs  
APPENDIX 2    PROJECT SAMPLING SOPs



**Title:** DRAFT QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 1 of 39

**Form A**  
**Title and Approval Page and Introduction**

Quality Assurance Project Plan for Vapor Intrusion Assessment  
Document Title

Prepared by: Anne Sheehan, GeoTrans, Inc.  
Prepared by: (Preparer's Name and Organizational Affiliation)

One Monarch Drive, Littleton, Massachusetts 01460, 978-952-0120  
Address and Telephone Number

Project Manager: *Anne Sheehan*  
Signature

Anne Sheehan  
Printed Name/Date

Project QA Officer: *Deborah Gaynor*  
Signature

Deborah Gaynor 3/25/10  
Printed Name/Date

USEPA Project Manager Approval: \_\_\_\_\_  
Signature

Joseph F. LeMay  
Printed Name/Date

USEPA Quality Assurance Approval: \_\_\_\_\_  
Signature

\_\_\_\_\_  
Printed Name/Date

**Title:** DRAFT QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 2 of 39

## **Form A (continued)**

### **Title and Approval Page and Introduction**

This Quality Assurance Project Plan (QAPP) has been prepared on behalf of UniFirst Corporation (UniFirst) and W.R. Grace & Co. – Conn (Grace) by GeoTrans, Inc. for submittal to the United States Environmental Protection Agency Region 1 (USEPA). This QAPP documents the groundwater sampling, analytical, and data review techniques that will be implemented during the Vapor Intrusion Assessment (VIA) that will be performed to assess the potential for off-property vapor intrusion from groundwater contamination in a portion of the Northeast Quadrant of the Wells G&H Superfund Site, as well as the groundwater sampling and analytical techniques that are part of the Indoor Air Quality and Vapor Intrusion Assessment (IAQA/VI) that will be performed at the UniFirst Property to assess the vapor intrusion potential on the UniFirst property. All work under this QAPP will be conducted within the State of Massachusetts, which is located in EPA Region 1.

This QAPP documents proposed monitoring well installation, well integrity testing, and groundwater sampling activities to be implemented during the VIA and IAQA/VI investigations. This QAPP describes the quality assurance/quality control (QA/QC) procedures to be used during the investigations to meet data quality objectives.

As discussed with USEPA in an in-person meeting on January 21, 2010, the expedited schedule and scope of the VIA and IAQA/VI investigations are consistent with preparation of a streamlined QAPP for this task. This QAPP has been prepared in general accordance with a March 2009 QAPP for the Norwood PCB Superfund Site Redevelopment provided by the USEPA as an example of an acceptable simplified QAPP format that includes all of the elements required by the EPA QA/G-5 “Guidance for Quality Assurance Project Plans” as well as the QAPP prepared by The Johnson Company for the IAQA/VI at the UniFirst Property (The Johnson Company, 2010a).

This QAPP is submitted to the USEPA Project Manager for review. No sample collection work will be conducted without prior approval by GeoTrans’ Project Manager and Quality Assurance Officer, the USEPA Quality Assurance Officer, the USEPA Project Manager, UniFirst and Grace, as applicable. Procedures described in this QAPP will be routinely followed, unless modifications and/or additions are documented in separate addenda or modification documents.

**Title:** DRAFT QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 3 of 39

### **Distribution List**

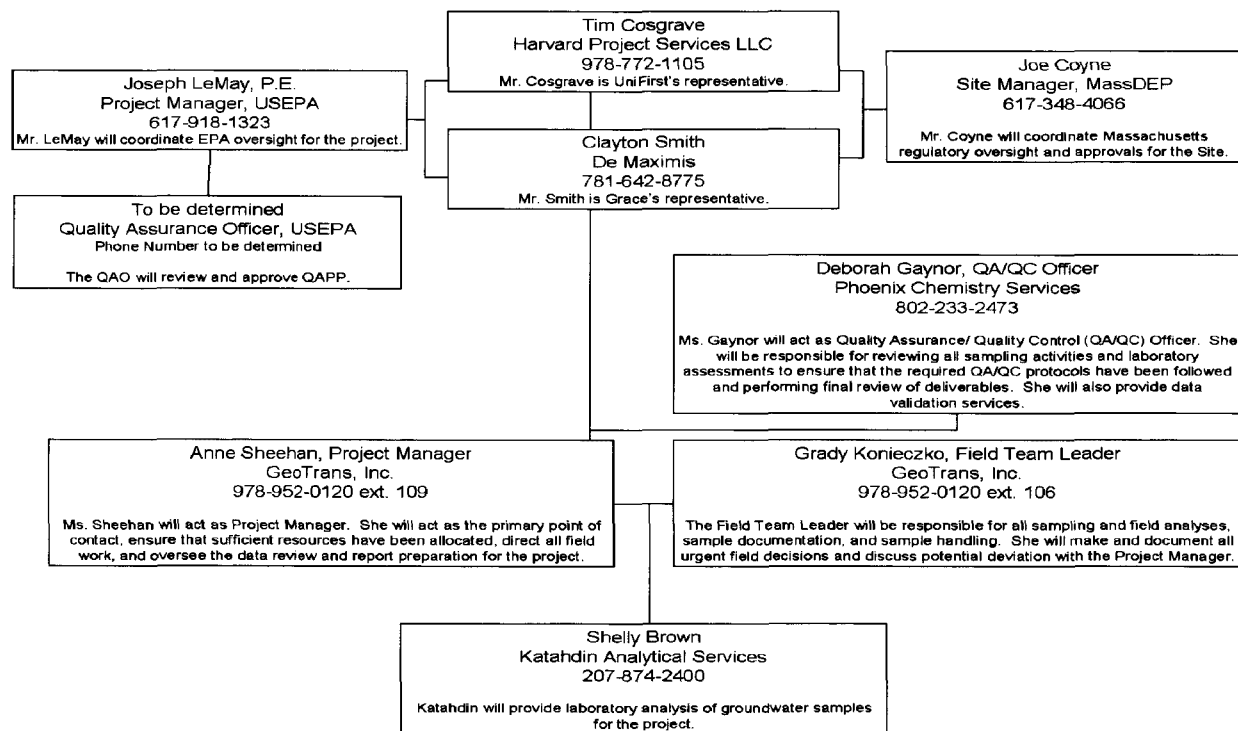
Names and telephone numbers of those receiving copies of this QAPP:

Joseph LeMay	USEPA	617-918-1323
Joseph Coyne	MassDEP	617-348-4066
Tim Cosgrave	Harvard Project Services LLC	978-772-1105
Clayton Smith	de maximis, inc.	781-642-8775
Robert Medler	Remedium Group, Inc.	901-820-2024
Jay Bridge	GeoTrans, Inc.	518-695-3092
Anne Sheehan	GeoTrans, Inc.	978-952-0120
Jack Guswa	JG Environmental	978-266-2992
Michael Moore	The Johnson Company	802-229-4600
Bettina Longino	The Johnson Company	802-229-4600
Deborah Gaynor	Phoenix Chemistry Services	802-233-2473
Shelly Brown	Katahdin Analytical Services	207-874-2400

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 4 of 39

## Form B Project Organization and Responsibility



**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 5 of 39

## **Form C**

### **Problem Definition/Background**

The Wells G&H Superfund Site (Site) in Woburn, Massachusetts, is bounded on the north by Route 128 (Interstate route 95), on the east by Interstate Route 93, on the south by Cedar and Salem Streets, and on the west by the MBTA railroad tracks. The Record of Decision (ROD) identified three operable units: OU1 which consists of five source area properties, including the UniFirst and Grace properties; OU2, the Central Area, and OU3 the Aberjona River, its tributaries, and their sediment and associated wetlands. The Central Area includes the entire Site with the exception of the five ROD-named source area properties and OU3.

In 1992 UniFirst and Grace implemented groundwater remedies for the UniFirst and Grace properties. The currently operating remedies include a 200-foot deep bedrock extraction well on the UniFirst property and sixteen recovery wells in the unconsolidated deposits and shallow bedrock on the Grace property. Extracted groundwater is treated at treatment plants located on the UniFirst and Grace properties. The coordinated groundwater remedies have been operating for more than 17 years. Monitoring data demonstrate that the coordinated remedies have established a widespread capture zone. Operation of the remedies has resulted in a substantial reduction in groundwater contaminant concentrations at the properties and throughout the Northeast Quadrant.

As USEPA stated in its 2004 Five-Year Review (USEPA, 2004), the potential vapor intrusion pathway in this portion of the Site has been evaluated repeatedly in the past 20 years. The area was the subject of indoor air sampling in July 1989 and October 1991, followed by an evaluation of those data in 1995 (ATSDR, 1995). In 1989, 1991, and 1995, USEPA and ATSDR concluded that the vapor intrusion pathway presented no apparent public health hazard. USEPA reexamined the historical data in 2004 and reached the same conclusion based on then current toxicological information. In 2004 USEPA also conducted vapor intrusion modeling using then current groundwater contaminant concentrations, measured as part of the long-term monitoring programs instituted by Grace and UniFirst in the Northeast Quadrant. USEPA concluded in 2004 that estimated risks based on its modeling results "are within or below EPA risk management guidelines, confirming earlier results based on indoor air sampling" (USEPA, 2004). Despite these findings USEPA recommended the installation of additional monitoring wells in the Northeast Quadrant to collect groundwater data to evaluate the vapor intrusion pathway.

On May 14, 2009, USEPA provided Grace and UniFirst with draft Comments on the OU-1 Grace and UniFirst Remedial Action Reports and the 1994 Central Area Remedial Investigation Phase 1A Report (Comments). The Comments letters requested work plans to re-evaluate potential vapor intrusion risks within the portion of the Central Area near the UniFirst and Grace properties, as well as to evaluate vapor intrusion risks in the building located on UniFirst property. In subsequent discussions with USEPA concerning its Comments, UniFirst

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 6 of 39

**Form C (continued)**  
**Problem Definition/Background**

and Grace agreed to submit a scope of work (SOW) for a Vapor Intrusion Assessment to collect groundwater samples to assess potential for vapor intrusion within the Northeast Quadrant. UniFirst also submitted a scope of work for the IAQA/VI at the UniFirst Property (The Johnson Company, 2010b). This QAPP details the QA/QC procedures to be used during the installation of groundwater monitoring wells, well integrity testing and collection of groundwater samples that are part of those investigations.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 7 of 39

## **Form D**

### **Project Description/Timeline**

The purpose of the VIA and IAQA/VI investigations are to collect groundwater samples to assess the potential for vapor intrusion from groundwater contamination in a portion of the Northeast Quadrant of the Wells G&H Superfund Site and on the UniFirst property. Grace and UniFirst have designed these investigations to address the USEPA request related to evaluating the potential for vapor intrusion in the study area by determining if VOCs are present in the shallow groundwater at sufficient concentrations to pose a vapor intrusion risk to nearby structures.

To collect the information to assess the potential for vapor intrusion, the following work is proposed:

- Install two shallow monitoring wells west of the UniFirst property;
- Install nine shallow monitoring wells in the Dewey Avenue neighborhood;
- Install two shallow monitoring wells south of the Grace property;
- Conduct well integrity tests of existing monitoring wells proposed for sampling that are not included in the Grace and UniFirst Northeast Quadrant remedy long-term monitoring programs; and
- Collect groundwater samples for VOC analyses from:
  - 13 newly installed wells;
  - Seven existing water table groundwater monitoring wells located south of the Dewey Avenue neighborhood; and
  - Existing, useable on-property water-table groundwater monitoring wells on the UniFirst property.

A comprehensive analyte list (see Form K) will be used for laboratory analysis of groundwater samples collected in association with the VIA and IAQA/VI. This list includes all VOCs where the maximum concentration detected in groundwater in monitoring wells located on either the Grace or UniFirst properties was greater than the current Screening Criteria proposed by USEPA (USEPA, 2010) or where the previous detection limits were greater than the Screening Criteria proposed by USEPA. All samples collected will be analyzed at Katahdin Analytical Services (Katahdin), Scarborough, Maine. Katahdin is a National Environmental Laboratory Accreditation Program (NELAP)-certified laboratory.

Proposed locations for the new monitoring wells are presented on Figure 1. Note that the proposed new monitoring well locations are approximate and may be adjusted depending on

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 8 of 39

### **Form D (continued)** **Project Description/Timeline**

accessibility. Locations of existing water-table monitoring wells to be sampled are also shown on Figure 1.

The first round of groundwater sampling will begin approximately two weeks after development of the new monitoring wells is complete. A second round of groundwater sampling will be performed approximately six months later. The groundwater samples will be collected using a low-flow sampling methodology. The pump intake will be located within the well screen, approximately two feet below the water level in the well and at least one foot below the top of the well screen. In cases where the well yield is insufficient to collect a sample using the low-flow sampling method, samples will be collected using a bailer.

All data collected during the VIA and IAQA/VI investigations will be summarized in a tabular format and presented in the VIA and IAQA/VI findings reports. Electronic tables of validated data also will be included with the findings reports. The findings reports will also include a summary of the work conducted and the data validation performed.

#### **Project Timeline**

The proposed project timeline is summarized below.

<b>Activity</b>	<b>Activity Start</b>	<b>Activity End</b>
USEPA QAPP review and approval (1.5 weeks)	3/25/10	4/7/10
Coordination of access for drilling (4 weeks)	3/17/10	4/14/10
Monitoring well installation and development (2 weeks)	4/15/10	4/30/10
Groundwater sampling, round 1 (1 week)	5/13/10	5/21/10
Laboratory analysis (3 weeks)	5/24/10	6/11/10
Data validation and evaluation (5 weeks)	6/14/10	7/16/10
Groundwater sampling, round 2 (1 week)	11/22/10	11/30/10
Laboratory analysis (3 weeks)	12/1/10	12/21/10
Data validation and evaluation (5 weeks)	12/22/10	1/25/11
VIA report preparation (5 weeks)	1/19/11	3/1/11
VIA report submitted to USEPA	3/2/11	3/2/11



**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 9 of 39

## **Form E**

### **Sampling Design**

Well integrity testing, monitoring well installation, and groundwater sampling will be performed according to GeoTrans' Standard Operating Procedures (SOPs) included in Appendix 2. No subcontracted field services will be required to conduct the VIA or IAQA/VI. All groundwater samples will be analyzed at Katahdin using the analytical method specified in Form F for the analyte list specified on Form K. The analytical methods used by the laboratory are described in their SOPs, attached in Appendix 1.

#### **Monitoring Well Installation**

To assess the potential for vapor intrusion in the study area, 13 2-inch diameter shallow monitoring wells will be installed in the unconsolidated deposits. The proposed well locations are shown on Figure 1. The wells will be drilled to a depth of approximately ten feet below the water table. It is anticipated that monitoring well depths will be 15 to 30 feet below ground surface. Drilling will be conducted using roto sonic and/or rotary techniques. Two-inch diameter polyvinyl chloride (PVC) wells with ten feet of ten-slot screen will be installed such that the water table is within the screened interval of the well. The wells will be completed to grade with flush-mount well caps. Well development will take place at the time of installation. All development water will be contained and treated at the Grace or UniFirst groundwater treatment system. Detailed procedures for well installation and development are presented in Appendix 2.

All proposed monitoring wells within the Dewey Avenue neighborhood will be installed on City of Woburn property. The proposed wells south of the Grace property and west of the UniFirst property, will be installed on Cummings property. Appropriate local permits, access agreements, and police detail, if required, will be arranged prior to initiating work conducted in or near public roadways or on public property.

#### **Well Integrity Testing**

Prior to collecting samples from existing monitoring wells not included in the Grace and UniFirst Northeast Quadrant remedy long-term monitoring programs (e.g., GO1S, K55S, K60S, S21, S22, and S63S and UniFirst wells S70S, UC16, UC17, UC19S, UC20, UC24S, UC25, UC26S, UC29S, UC30, UC31S, UC32, UC33, UC34, UC35, UC36, UC4, UC5, and UC8), well integrity tests will be conducted. These tests will include a visual inspection of the well, depth measurement to confirm well depth, and a slug test to confirm the hydraulic connection of the well to the saturated unconsolidated deposits. If the well integrity tests indicate that well development is necessary, the wells will be redeveloped by bailing. These wells will not be sampled if the well integrity tests indicate that the wells are not usable. The well locations are shown on Figure 1. Locations on Figure 1 are well cluster names and do not always indicate the "S" suffix; for instance, UC24S is shown on the figure as UC24. Detailed procedures for well integrity testing are presented in Appendix 2.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 10 of 39

## **Form E (continued)**

### **Sampling Design**

#### **Groundwater Sampling**

Two rounds of groundwater samples will be collected from the new monitoring wells, seven existing water table groundwater monitoring wells located south of the Dewey Avenue neighborhood, and existing useable on-property water-table groundwater monitoring wells on the UniFirst property. The wells proposed for sampling are shown on Figure 1. In order to collect samples that are representative of the groundwater quality at the water table, the sampling will be performed using the low-flow groundwater sampling methodology described in Appendix 2. Groundwater sampling will begin approximately two weeks after well development is completed. The groundwater samples will be sent to a laboratory for analysis of the VOC analytes listed in Form K.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 11 of 39

## Form F

### Sampling and Analytical Methods Requirements

Sample Matrix	Parameter	Number of Samples (including field QC samples)	Analytical Method (SOP)*	Sampling SOP*	Sample Containers (number per sample, size, and type)	Sample Preservation (temperature, light, chemical)	Maximum Holding Time
Groundwater Round 1	VOCs-Laboratory	42 samples 4 trip blanks** 3 field duplicates 2 rinsate blanks*** 3 MS/MSD	SW-846 5030/8260B (Full Scan and SIM) Katahdin Analytical Services SOPs (CA-202, CA-220)	3	3-40 mL VOC vials, Teflon-lined septa	HCl to pH <2, 4 $\pm$ 2 °C, no headspace	14 days to analysis
Groundwater Round 2	VOCs-Laboratory	42 samples 4 trip blanks** 3 field duplicates 2 rinsate blanks*** 3 MS/MSD	SW-846 5030/8260B (Full Scan and SIM) Katahdin Analytical Services SOPs (CA-202, CA-220)	3	3-40 mL VOC vials, Teflon-lined septa	HCl to pH <2, 4 $\pm$ 2 °C, no headspace	14 days to analysis

**Notes:**

- \* Form G contains method and SOP reference tables.
- \*\* Estimated. One trip blank per cooler with VOC samples.
- \*\*\*Estimated. One rinsate blank per equipment type per sampling round.

**Abbreviations:**

VOC = Volatile Organic Compound  
 QC = Quality Control  
 SOP = Standard Operating Procedure  
 SIM = selective ion monitoring  
 MS/MSD = Matrix Spike/Matrix Spike Duplicate

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 12 of 39

**Form G**  
**Method and SOP Reference Table**

<b>Analytical Method Reference<sup>1</sup>:</b> <b>Includes document title, method name/number, revision number, and date</b>	
<b>No.</b>	<b>Analytical Method Name</b>
1	EPA Method SW-846 8260B, Volatile Organic Compounds Determined by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

<sup>1</sup> Analytical method is referenced in the laboratory's SOP.

<b>Project Analytical Standard Operating Procedures (SOPs)<sup>2</sup>:</b> <b>Includes document title, date, revision number, and originator's name</b>	
<b>No.</b>	<b>Analytical SOP Name</b>
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, Revision 11, 08/09, Katahdin Analytical Services, Inc.
CA-220	Analysis Of Volatile Organic Compounds By Purge And Trap GC/MS SW-846 Method 8260 – Modified For Selected Ion Monitoring (SIM), Revision 7, 08/09, Katahdin Analytical Services, Inc.
SD-902	Sample Receipt and Internal Control, SOP No. SD-902, Revision 8, 08/09, Katahdin Analytical Services, Inc.
SD-903	Sample Disposal, SOP No. SD-903, Revision 4, 05/09, Katahdin Analytical Services, Inc.

<sup>2</sup> SOP is included in Appendix 1.

<b>Project Sampling SOPs<sup>3</sup>:</b> <b>Includes document title, revision number, and originator's name</b>	
<b>No.</b>	<b>Sampling SOP Name</b>
1	Standard Operating Procedure for Well Integrity Testing, Rev. 03/10, GeoTrans, Inc.
2	Standard Operating Procedure for Monitoring Well Installation, Rev. 03/10, GeoTrans, Inc.
3	Standard Operating Procedure for Groundwater Sampling, Rev. 03/10, GeoTrans, Inc.
4	Standard Operating Procedure for Materials Management, Rev. 03/10, GeoTrans, Inc.

<sup>3</sup> SOP is included in Appendix 2.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 13 of 39

**Form H**  
**Field Equipment Calibration and Corrective Action**

<b>Instrument</b>	<b>Activity</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>SOP Reference*</b>
MiniRAE 3000 PID	Visual Inspection  Calibration (beginning of day)/ Verification (end of day)	Beginning and end of day, more often if response is slow	Manufacturer's Guidance	Clean lamp and unit; re-zero and recalibrate with 100 ppm isobutylene. Replace if necessary.	SOP 2, Manufacturer's Guidance
YSI 556	Visual Inspection  Calibration (beginning of day)/ Verification (end of day)	Beginning and end of day, more often if response is slow.	Manufacturer's Guidance	Clean probes and unit; re-zero and recalibrate. Replace if necessary.	SOP 3, Manufacturer's Guidance
Turbidity Meter	Visual Inspection  Calibration (beginning of day)/ Verification (end of day)	Beginning and end of day, more often if response is slow.	Manufacturer's Guidance	Clean probes and unit; re-zero and recalibrate. Replace if necessary.	Manufacturer's Guidance
Water Level Meter	Visual Inspection  Field checks as per manufacturer	Daily  Once upon receiving from vendor	Factory-determined	Replace if necessary.	Manufacturer's Guidance

\*Form G contains the SOP reference table.

Abbreviation:

ppm = part per million

NTU = nephelometric turbidity units

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 14 of 39

**Form H (continued)**  
**Inspection/Acceptance Requirements for Supplies and Consumables**

<b>Supplies</b>	<b>Inspection Frequency</b>	<b>Type of Inspection</b>	<b>Responsible Party</b>	<b>Corrective Action</b>
Calibration gas for PID	During mobilization preparation	Verify positive pressure in canister	Sampler	Replace or fill canister
Calibration solutions for YSI	During mobilization preparation	Verify quantity and expiration dates of new calibration solutions	Sampler	Replace calibration solutions
Decontamination solutions	During mobilization preparation	Verify quantity of methanol and liquinox is available	Sampler	Purchase additional decontamination solutions
De-ionized water	During mobilization preparation	Verify quantity of DI water is available	Sampler	Purchase additional DI water
PID headspace jars	During mobilization preparation	Ensure adequate quantity of clean jars is available	Sampler	Purchase additional jars
Bladder / Grundfos / Peristaltic pumps	During mobilization preparation	Verify equipment is in working condition	Sampler	Replace or repair defective equipment
Generator	During mobilization preparation	Verify equipment is in working condition	Sampler	Replace or repair defective equipment or parts
Compressed air cylinders	During mobilization preparation	Ensure adequate volume of compressed air is present for low flow sampling	Sampler	Rent additional compressed air cylinders
Poly tubing	During mobilization preparation	Ensure adequate quantity and appropriate diameter tubing is available	Sampler	Purchase additional poly tubing
Sampler connections and compressed gas regulators/fittings	During mobilization preparation	Verify the number and types of connections are correct	Sampler	Obtain additional connections and/or fittings

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 15 of 39

**Form H (continued)**  
**Inspection/Acceptance Requirements for Supplies and Consumables**

<b>Supplies</b>	<b>Inspection Frequency</b>	<b>Type of Inspection</b>	<b>Responsible Party</b>	<b>Corrective Action</b>
PPE	During mobilization preparation	Verify adequate quantity of nitrile gloves, ear and eye protection, hard hats, and other required PPE are available	Sampler	Purchase additional PPE as required
Drilling/Lithological record forms	During mobilization preparation	Verify that appropriate type and number of forms are available	Sampler	Obtain additional copies before sampling
Low Flow sampling forms and sample labels	During mobilization preparation	Verify that appropriate type and number of forms will accompany samples	Sampler	Obtain additional copies before sampling
Chains of custody for samples	During mobilization preparation	Verify that appropriate type and number of forms will accompany samples	Sampler	Obtain additional copies before sampling
VOA Vials	During mobilization preparation	Verify that appropriate type and number have been supplied by laboratory	Sampler	Obtain additional vials before sampling
Coolers/Ice	During daily mobilization preparation	Verify that necessary coolers and ice are available	Sampler	Obtain additional coolers/ice before sampling
Electronic data storage media	During use and upon saving data, before any paper data is destroyed	Verify that all files are stored on the local area network in the correct location	Project Manager	Move to correct location or re-save data in correct location

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 16 of 39

## Form I

### Laboratory Equipment Calibration and Corrective Action

Instrument	Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Ref*
GC/MS VOCs (Full Scan and SIM)	Tune	Every 12 hours.	Criteria listed in section 7.3 current revision of SOP CA-202 and CA-220.	Retune and/or clean source.	CA-202, CA-220
	Initial calibration	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria. Six-point initial calibration for all analytes.	SPCCs RF $\geq 0.050$ ; RSD $\leq 30\%$ for the CCCs; %RSD $< 15\%$ for all other compounds. If not met: Option 1) Linear least squares regression: $r \geq 0.995$ Option 2) Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	
	ICV (Second Source)	Once after each initial calibration.	$\pm 25\%$ recovery individual compounds.	Identify source of problem, correct, repeat calibration, rerun samples.	
	Continuing calibration verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune.	CCCs $< 20\%D$ (D = Difference or Drift); SPCCs RF $> 0.10$ & $0.30$  D = Difference or drift	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	

Notes:

\*Form G contains the method and SOP reference tables.

**Abbreviations:**

GC/MS = Gas chromatograph/mass spectrometer  
 ICV = Initial calibration verification  
 CCV = Continuing Calibration Verification  
 BFB = bromofluorobenzene  
 SPCC = System Performance Check Compounds  
 RSD = Relative Standard Deviation  
 CCC = Calibration Check Compounds  
 COD = Coefficient of Determination  
 D = Difference or Drift  
 RF = Response Factor



**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 17 of 39

## **Form J**

### **Sample Handling and Custody Requirements**

An established program of sample chain-of-custody procedures shall be followed during sample collection and handling activities in both the field and laboratory operations in order to assure that sample integrity is maintained and data generated by the analysis of the samples is applicable to a proper evaluation of the site. The objectives of sample identification, custody, and monitoring procedures are to assure that:

1. All samples collected are uniquely labeled for identification throughout the analytical process;
2. Samples are correctly analyzed and results are traceable to field records;
3. Important sample characteristics are preserved;
4. Samples are protected from loss, damage, or tampering;
5. Any alteration of samples (e.g., filtration, preservation, or damage due to shipment or other processes) is documented; and
6. A record of sample integrity and analytical fate is established for legal purposes.

The following sections describe the sample labeling, chain-of-custody forms, and packing and shipping requirements for the project.

#### **SAMPLING LABELING**

Each sample bottle will be labeled with a selected numerical or alphanumeric designation that allows the sampling team to identify the sample for tracking purposes and allows the technical staff to correlate the results of the vapor intrusion assessment with the ongoing monitoring effort.

Additional information to be shown on the bottle label will include:

1. General site name, identification, or location;
2. Sampling location, i.e., well number or location;
3. Sampling date and time;
4. Name of person collecting the sample;
5. Preservatives which have been added to the sample; and
6. The sample analysis to be performed.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 18 of 39

## **Form J (continued)**

### **Sample Handling and Custody Requirements**

#### **CHAIN-OF-CUSTODY RECORD**

Chain-of-custody records are used to ensure that samples are traceable from the time of collection until ultimate disposal. A sample is in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession;
2. The sample is in the person's view after being in possession;
3. The sample has been locked up to prevent tampering after it was in the person's possession;
4. The sample was in the person's possession and was then transferred to a designated secure area.

The chain-of-custody record is initiated in the field by the individual physically in charge of the sample collection. The chain-of-custody record may be completed contemporaneously with the sample data sheet or prior to the shipment of samples to the laboratory.

The sampler is personally responsible for the care and custody of the sample until it is transferred or dispatched properly. When transferring the possession of samples, the individuals relinquishing and receiving the sample will sign, date, and write the time on the chain-of-custody record. The chain-of-custody record contains information on the date/time of sample collection, the sample identification, the sampler, the project name and number, laboratory project number, the destination of the samples, the number of containers of each sample being shipped, type of preservation, sample matrix, QC sample designation, and an itemization of the analyses requested for each sample, as well as any remarks about the sample, including special handling instructions, the method of shipment, and courier's name.

The chain-of-custody record is enclosed with the samples after it has been signed and dated by the sampler. The top copy of the chain-of-custody form will be retained by the sampler. The rest of the chain-of custody form will be placed in a plastic (preferably Ziploc) bag, and sealed in the shipping container.

Laboratory custody procedures are addressed in the specific laboratory SOP (SD-902, Appendix 1).

All waste sample material will be disposed of by the analytical laboratory in accordance with all state and federal regulations (SD-903, Appendix 1).

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 19 of 39

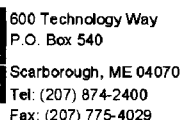
## **Form J (continued)**

### **Sample Handling and Custody Requirements**

#### **PACKING AND SHIPPING**

All samples will be packed in an insulated container with ice added immediately after sample collection. Ice cubes placed in plastic bags will be added as necessary to achieve an internal cooler temperature of 2 to 6 degrees Celsius. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The sample bottles will then be wrapped in bubble pads and placed upright in the cooler to cushion the samples to prevent breakage. Vermiculite and/or Styrofoam will be used as packing material. The cooler will be latched and sealed with strapping tape. A custody seal will be placed over the closed cooler and container lid to reveal any tampering. The coolers will be attended by GeoTrans personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory. The coolers will be labeled and shipped by courier or overnight carrier within 24 hours of collection to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding time before sample analysis is presented in Form F. Form F also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.


**Revision Number: 0**  
**Revision Date: 03/25/10**  
**Page: 20 of 39**



Client:		Contract:		Phone #: (    )		Fax #: (    )							
Address:		City:		State:		Zip Code:							
Purchase Order #:		Proj. Name/No.:		Katahdin Quote #:									
Bill (if different than above):				Address:									
Sampler (Print/Sign):				Copies To:									
LAB USE ONLY		Work Order # Katahdin Project Number		Analysis and Container Type Preservatives									
Remarks:				Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N	Filt. Y/N
Shipping Info:		FEDEX      UPS      CLIENT											
Airbill No.													
Temp C		Temp Blank      Intact      Not Intact											
*	Sample Description	Date/Time Collected	Matrix	No. of Containers									
COMMENTS:													
Relinquished By:		Date/Time	Received By:		Relinquished By:		Date/Time		Received By:				
Relinquished By:		Date/Time	Received By:		Relinquished By:		Date/Time		Received By:				

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 21 of 39

 **GeoTrans, Inc.** Label 1 of 1  
SITE: \_\_\_\_\_ MATRIX: \_\_\_\_\_  
SAMPLE\_ID: \_\_\_\_\_ PRESRV: \_\_\_\_\_  
ANALYSES: \_\_\_\_\_  
DATE: \_\_/\_\_/\_\_ TIME: \_\_:\_\_ SAMPLER: \_\_\_\_\_

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 22 of 39

## Form K

### Analytical Sensitivity and Project Criteria

Analyte	Analytical Method <sup>1,2</sup>	Detection Limit (µg/L)	Reporting Limit (µg/L)	Precision Difference (%)	Accuracy Limit (%)	Proposed Screening Level <sup>3</sup> (µg/L)
<i>Laboratory VOC Analysis (Groundwater)- SOP CA-202, CA-220</i>						
1,1,1-Trichloroethane	SW-846 8260B	0.20	1.00	0-30	77-129	714
1,1,2-Trichloroethane	SW-846 8260B	0.33	1.00	0-30	84-115	4.11
1,1-Dichloroethane	SW-846 8260B	0.21	1.00	0-30	76-130	6.61
1,1-Dichloroethene	SW-846 8260B	0.35	1.00	0-30	88-127	18.7
1,2,4-Trimethylbenzene	SW-846 8260B	0.19	1.00	0-30	83-118	2.8
1,2-Dibromoethane	SW-846 8260B SIM	0.019	0.05	0-30	70-130	0.135
1,2-Dichloroethane	SW-846 8260B	0.20	1.00	0-30	81-125	2.34
1,2-Dichloropropane	SW-846 8260B	0.25	1.00	0-30	84-118	2.12
1,3-Dichlorobenzene	SW-846 8260B SIM	TBD	≤0.50	0-30	70-130 (nominal)	NA*
1,4-Dichlorobenzene	SW-846 8260B	0.24	1.00	0-30	86-111	2.25
Benzene	SW-846 8260B	0.26	1.00	0-30	86-116	1.36
Bromodichloromethane	SW-846 8260B SIM	0.016	0.05	0-30	70-130 (nominal)	0.94
Bromoform	SW-846 8260B	0.23	1.00	0-30	86-117	101
Carbon tetrachloride	SW-846 8260B SIM	0.016	0.05	0-30	70-130 (nominal)	0.135
Chlorobenzene	SW-846 8260B	0.22	1.00	0-30	89-113	33.3
Chloroform	SW-846 8260B SIM	0.05	0.1	0-30	70-130 (nominal)	0.705
cis-1,2-dichloroethene	SW-846 8260B SIM	TBD	≤0.50	0-30	70-130 (nominal)	NA*
Ethylbenzene	SW-846 8260B	0.21	1.00	0-30	88-113	3.04
Isopropylbenzene	SW-846 8260B SIM	TBD	≤0.50	0-30	70-130 (nominal)	0.844
Methylene chloride	SW-846 8260B	1.13	5.00	0-30	72-129	57.5
Naphthalene	SW-846 8260B	0.30	1.00	0-30	62-126	3.98
Tetrachloroethene	SW-846 8260B SIM	0.011	0.05	0-30	70-130 (nominal)	0.55
Toluene	SW-846 8260B	0.27	1.00	0-30	84-118	1850
trans-1,2-dichloroethene	SW-846 8260B	0.25	1.00	0-30	78-125	15.8
trans-1,3-Dichloropropene	SW-846 8260B SIM	0.011	0.05	0-30	70-130 (nominal)	0.84
Trichloroethene	SW-846 8260B	0.28	1.00	0-30	79-121	2.89
Vinyl chloride	SW-846 8260B SIM	0.01	0.05	0-30	70-130	0.145
Xylenes (total)	SW-846 8260B	0.25	3.00	0-30	89-116	35.7

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 23 of 39

**Form K (continued)**  
**Analytical Sensitivity and Project Criteria**

**Notes:**

<sup>1</sup> Form G contains the method and SOP reference tables.

<sup>2</sup> Samples will be analyzed using Method SW-846 8260B, with SIM where necessary and possible to achieve applicable reporting limits.

<sup>3</sup> EPA Vapor Intrusion Screening Criteria provided in 2/25/10 letter (USEPA, 2010)

\*While no screening criterion has been established for this target compound, the laboratory should achieve a reporting limit of 0.5 µg/L.

**Abbreviations:**

µg/L = micrograms per liter

SIM = Selective Ion Monitoring

NA = Not applicable

TBD = To be determined (Values currently being determined; will be supplied prior to groundwater sampling.)

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 24 of 39

## Form L Field Quality Control

QC Sample	Frequency	Acceptance Criteria	Corrective Action(s)
Trip Blank	Ship 1 trip blank per cooler containing VOC samples.	No reported VOC concentrations at or above the quantitation limits	<p>The occurrence will be reviewed to address possible procedural changes to eliminate the source of contamination.</p> <p>The affected positive sample results below 5x the blank concentration (10x the blank concentration for common laboratory contaminants) will be changed to non-detected values (U) in accordance with the Region I Data Validation Guidelines.</p>
Field Duplicate	One per 20 field samples.	RPD $\leq$ 30%. If sample results are $< 2 \times \text{QL}$ , professional judgment is used.	The affected positive and non-detected results will be estimated (J, UJ) in original sample and duplicate sample in accordance with the Region I Data Validation Guidelines.
Rinsate Blank	One per equipment type per sampling round.	No target analytes $\geq \frac{1}{2} \text{QL}$ ( $> \text{QL}$ for common laboratory contaminants), unless target analytes in field samples are $> 10 \times$ those in rinsate blank.	The affected positive sample results below 5x the blank concentration (10x the blank concentration for common laboratory contaminants) will be changed to non-detected values (U) in accordance with the Region I Data Validation Guidelines.
Temperature Blank	One per cooler.	Temperature between 2 and 6 degrees Celsius ( $4 \pm 2^\circ \text{C}$ ).	<p>In the affected samples, the positive results will be estimated (J) and the affected non-detected results will be rejected (UR) in accordance with the Region I Data Validation Guidelines.</p> <p>If the temperature slightly exceeds 6 deg C, or is slightly below 2 deg C, professional judgment will be used to qualify or not qualify the data.</p>

Abbreviations:

RPD = Relative percent difference (Equation for the calculation of RPD is provided in Form R)

QL = Quantitation Limit



**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 25 of 39

## Form M Laboratory Quality Control

QC Item	Frequency	Methodology	Acceptance Criteria	Corrective Action(s)
Lab Control Sample (LCS)	One per batch of 20 or less.	SW846 8260 / SW846 8260 - SIM	Recovery must be within Katahdin Analytical Services' statistically-derived limits.	Evaluate and reanalyze if possible. If a matrix spike/matrix spike duplicate (MS/MSD) was performed in the same 12 hour clock and acceptable, narrate. If the LCS recoveries are high but the sample results are <QL narrate, otherwise re-prepare and reanalyze.
Laboratory Blank	One per batch of 20 or less.	SW846 8260 / SW846 8260 - SIM	No target compounds should be > the QL except common lab contaminants, which should be < the 2 x QL.	Investigate source of contamination. Rerun method blank prior to analysis of samples if possible. Evaluate the samples and associated QC: if blank results are above QL, report sample results which are < QL or > 10X the blank concentration. Reanalyze blank and samples >QL and < 10X the blank.
Internal Standard (IS)	Three per sample- Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4	SW846 8260 / SW846 8260 - SIM	Retention times for internal standards must be $\pm 30$ seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples that do not meet IS criteria or were analyzed while system was malfunctioning.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 03/25/10  
**Page:** 26 of 39

### Form M (continued) Laboratory Quality Control

QC Item	Frequency	Methodology	Acceptance Criteria	Corrective Action
Surrogates	Four per sample	SW846 8260 / SW846 8260 - SIM	Percent recoveries (both methods): Dibromofluoromethane: 70-130 1,2-dichloroethane-d4: 70-130 Toluene-d8: 70-130 Bromofluorobenzene 70-130	If sample volume available and within hold time, reanalyze.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	SW846 8260 / SW846 8260 - SIM	Recovery should be within Katahdin Analytical Services' statistically-derived limits.  Water Precision: $RPD \leq 30\%$	Corrective actions will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, re- prepare the samples and QC.
Holding Times	Every sample	SW846 8260 / SW846 8260 - SIM	Analyze within 14 days of sampling	Contact client concerning any missed hold times.

## **Form N**

### **Data Management and Documentation**

Data management and sample tracking tasks are performed in accordance with GeoTrans SOP 1 through 4 (Appendix 2). Documentation to be used in the field investigation is described below.

#### **SITE LOG BOOK**

A bound site logbook (notebook) will be maintained by the Field Operations Leader (FOL). The FOL or designee will record all information related to sampling or field activities. This information will include sample time, weather conditions, unusual events, and field measurements. The site logbook will contain a summary of daily activities, and will reference field forms when applicable. The applicable field forms for this project are presented in GeoTrans' SOPs, which are included in Appendix 2.

#### **WELL INTEGRITY TEST LOGS**

Observations made during well integrity testing will be recorded on Well Integrity Test Log sheets. Information to be recorded on Well Integrity Test Logs will include historic and current monitoring well depth, description of condition of monitoring well, slug information and water level measurements. An example Well Integrity Test Log is presented in GeoTrans SOP 1 (Appendix 2).

#### **FIELD BORING LOGS**

Observations made during monitoring well installation will be recorded on Field Boring Log sheets. Information to be recorded on Field Boring Logs will include sample depth, PID field screening results, geological description of subsurface materials encountered, and any other pertinent observations about soil moisture condition, odors, or discoloration.

The construction details of all new monitoring wells will be documented on the Field Boring Log sheet. This log sheet will identify the total depth of the monitoring well, the depth of the screened interval, and provide a cross-section of the depths and types of materials utilized to complete the monitoring well. An example Field Boring Log data sheet is presented in GeoTrans SOP 2 (Appendix 2).

#### **GROUNDWATER SAMPLE COLLECTION RECORDS**

Groundwater sampling data specific to each monitoring well will be recorded on Low Flow Groundwater Sample Collection Record data sheets. The recorded information will include, at a minimum, date and time sampled, depth of pump intake, field water quality measurements, sample bottles filled, and sample preservations. An example Low Flow Groundwater Sample Collection Record data sheet is GeoTrans SOP 3 (Appendix 2).

**Form N (continued)**  
**Data Management and Documentation**

**PACKING LIST/CHAIN-OF-CUSTODY RECORD AND CUSTODY SEAL**

Please see description in Form J.

**LABORATORY SAMPLE LOG-IN PROCEDURE**

Custody Seals are supplied with all bottle orders. They are affixed to the cooler after sampling. The presence or absence of Custody Seals is noted on the Sample Receipt Condition Report (SRCR) described below.

Upon receipt of samples from the field, the laboratory sample management personnel will sign off on the Chain-of-Custody (COC), open the sample cooler(s), verify sample integrity, and conduct a check against the COC. If there is a discrepancy or problem (i.e. broken sample containers) the laboratory will contact the field leader or other field personnel and resolve the issue. Additionally, the laboratory completes a Sample Receipt Condition Report, which documents visual inspection of the samples and specific parameters such as cooler temperature, holding times, and preservation. Discrepancies or changes will be documented on the Sample Receipt Condition Report.

The laboratory sample management personnel assigns a unique laboratory work order number for the entire sample set listed on the chain of custody. The samples are then logged into the laboratory information system (KIMS) and a Login Chain of Custody Report is generated. Each sample within a work order is labeled numerically. Each container of a particular sample is uniquely identified by adding an alphabetical suffix to the sample number. The laboratory labels each sample container with a Laboratory Custody Label which will remain on the sample bottle for the duration of the laboratory sample storage. The laboratory also initiates the appropriate Internal Custody Record for the sample set. Personnel fill out the Internal Custody Records to document sample removal from and return to sample storage. A laboratory data file is also initiated for the work order. This file includes the Login Chain-of-Custody, Chain-of-Custody, and Sample Receipt Condition Report. The folder also includes a Login File Sheet which summarizes the analyses that the work order has been logged for. This sheet is used to track data completion.

Samples for a project may be batched or grouped together by the laboratory. A series of batched work orders is referred to as a sample delivery group (SDG). The SDG includes those samples received on a chain of custody, duplicate samples, and field QA/QC samples, and can include samples of different media. QA/QC samples will be run at the frequency specified in the analytical methods. The sample delivery group is given a specific identification number.

Samples are stored at the laboratory in refrigerators prior to, during, and after analysis. Refrigerators at the laboratory are constantly monitored for temperature. Proper temperatures and lighting are maintained in the refrigerators to ensure sample integrity and preservation. Samples are retained by the laboratory for a period of 90 days after the data report is mailed to

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 29 of 39

## **Form N (continued)**

### **Data Management and Documentation**

the client unless otherwise specified in a client contract. The laboratory then disposes of non-hazardous samples, following certified disposal practices. Hazardous samples are either returned to the client or disposed of through a licensed broker. Documentation of disposal is maintained by the laboratory.

Please refer to Katahdin Analytical Services SOPs SD-902 "Sample Receipt and Internal Control" and SD-903, "Sample Disposal" for more information (Appendix 1).

### **LABORATORY DATA PACKAGE**

The following outlines the components of the laboratory data package:

1. Sample Data Package
  - a. Narrative
  - b. Supporting Documents
  - c. Chain of Custody Record
  - d. Login Report
2. Sample Data Summary
  - a. Report of Analytical Results
3. Volatile Data
  - a. QC Summary
  - b. Sample Data
  - c. Standards Data
  - d. Raw QC Data
  - e. Logbooks and Supporting Documents

The Laboratory Data Report will be delivered by e-mail or electronic download in a PDF format.

### **ELECTRONIC DATABASE**

Data will be received by e-mail or electronic download directly from the laboratory in an Excel spreadsheet. The data will be added to the Site analytical database maintained by GeoTrans.

## **Form O**

### **Assessments and Response Actions**

#### **FIELD TASKS**

The field team is responsible for completing all field tasks in accordance with the Wells G&H Superfund Site Vapor Intrusion Assessment Work Plan, Revision 1 (GeoTrans, 2010), IAQA/VI Work Plan, Revision 2 (The Johnson Company, 2010b) and specified SOPs. The Field Operations Leader (FOL) is responsible for understanding the Work Plan objectives, and checking the completeness of each field task on a daily basis and upon completion of the field activities.

Changes in field operating procedures may be necessary as a result of changed field conditions or unanticipated events. A summary of the sequence of events associated with field changes is as follows:

1. If a substantial change is required, the FOL or designee notifies the GeoTrans Project Manager of the need for the change;
2. If necessary, the Project Manager will discuss the change with the client, USEPA MassDEP, or other stakeholders and will provide verbal approval or denial to the FOL or designee for the proposed change;
3. The FOL will document the change on a Field Corrective Action/Deviation (FCAD) form (see below) and forward the form to the GeoTrans Project Manager at the earliest convenient time;
4. The Project Manager will sign the form and distribute copies to the GeoTrans Program Manager, Quality Assurance Officer, FOL, and the project file; and
5. A copy of the completed FCAD form will also be attached to the field copy of any affected documents.

#### **LABORATORY TASKS**

The analytical laboratory is responsible for completing all laboratory tasks in accordance with all methods and SOPs. The laboratory must maintain its NELAP certification throughout the course of the project.

Whenever the laboratory is changed or internal procedures are changed for the existing laboratory, the laboratory QC procedures must be resubmitted before further sampling and analysis is conducted. In all cases, the internal laboratory QC procedures will comply with the requirements of the appropriate EPA analytical methods.

#### **MODIFICATIONS TO QAPP**

All major modifications to this QAPP must have prior approval by the USEPA Project Manager.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 31 of 39

### Field Corrective Action/Deviation Form

Corrective Action/Deviation Form No. \_\_\_\_\_

Site \_\_\_\_\_ Project No. \_\_\_\_\_

Originator \_\_\_\_\_ Date \_\_\_\_\_

Person Responsible for Replying \_\_\_\_\_

Description of problem and when identified: \_\_\_\_\_

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State cause of problem if known or suspected: \_\_\_\_\_

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Sequence of Corrective Action/Deviation: \_\_\_\_\_

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Approved By: \_\_\_\_\_ Date: \_\_\_\_\_

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 32 of 39

## **Form P**

### **Project Reports**

A report detailing the VIA will be provided to USEPA within 60 days after receipt of the validated analytical data from the second groundwater sampling event. The report will include:

- Objectives of the VIA;
- An overview of the monitoring well installation;
- Details regarding the groundwater quality samples and results; and
- Conclusions and recommendations.

Maps will be included that depict the sampling locations. Field and laboratory analytical data will be tabulated. The appendices will include: laboratory narratives, field deviation reports, and field data collection forms that have not otherwise been manipulated or summarized, such as well integrity test forms and low flow groundwater sample collection records.

The final project report will be provided in the following format: the main body of the report, summary tables, graphics, and appendices will be provided in hardcopy and in electronic format on a CD to the USEPA, MassDEP, and the clients.



**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 33 of 39

## **Form Q**

### **Field Data Evaluation**

Verification of the actual sampling procedures used will be performed first by the FOL or sampler, and finally by the QA Officer. All field notes, sampling forms, and deviation forms will be reviewed for errors and omissions. The field team will be in contact with the Project Manager daily or at reasonable intervals to discuss sampling procedures/events to minimize the chance that errors go undetected.

The QA Officer will evaluate the field records for consistency and review all pertinent QC information on these records. The deviation reports will be reviewed for consistency with field records, and to determine if appropriate corrective actions have been completed, and the impact of these deviations on project goals will be determined. The field notes will be compared to the chain-of-custody documents and laboratory reports to assess whether all samples were sent to the laboratory and analyzed. A field data validation report will be included as an appendix.

Certain field notes, including sampling forms, will be included in the appendix of the final investigation report. All original field forms will be filed by GeoTrans and retained in accordance with Article XXVI of the Wells G&H Consent Decree entered in the matter of United States vs. Wildwood Conservation Corporation *et al* (Civil Action 91-11807-MA) entered 10/9/91.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 34 of 39

## **Form R**

### **Laboratory Data Evaluation**

To assure groundwater data meets all data quality objectives and are representative of Site conditions, a Tier III data validation will be conducted. The data validation will be performed by a subcontracted data validator from Phoenix Chemistry Services (PCS). Data will be qualified, as necessary, based on the QC criteria for VOCs by SW-846 8260B listed in Forms I, L, and M of this QAPP. *Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II*, December 1996 will be applied using these criteria. The results of the validation will be summarized in a memorandum and provided in the final report. The memorandum will include identification of the project and sample identification numbers, the analyses performed, the data validation parameters evaluated, and discussion of the parameters that did not meet the QC criteria and the effects, if any, on data usability.

The precision and accuracy of data will be routinely assessed to assure that data collected is representative of the site. Both statistical procedures and qualitative evaluations will be utilized to verify the usability of the data. The data will be assessed for accuracy, precision, and completeness by taking into account the overall project objectives, background data points, and quality control samples. Procedures for assessing precision, accuracy, and completeness are described below.

#### **PRECISION – RELATIVE PERCENT DIFFERENCE**

RPD will be calculated to assess the precision of duplicates by the equation shown below.

$$RPD = \frac{X_1 - X_2}{X_{avg}} \times 100$$

Where:  $X_1$  = concentration of compound for Sample 1 of duplicate

$X_2$  = concentration of compound for Sample 2 of duplicate

$X_{avg}$  = average of Samples 1 and 2

If the RPD of field duplicates, matrix duplicates or lab duplicates, if used, is greater than 30 percent, the field techniques or laboratory techniques will be reviewed respectively.

## **Form R (continued)**

### **Laboratory Data Evaluation**

#### **ACCURACY – BLANKS**

Blank results will be used to assess the possibility of field or laboratory contamination and the usability of the sample data. All blank results including field blank, trip blank, and laboratory blank results will be reviewed to determine if any compounds were detected above the contract required quantitation limits (CRQL).

If compounds are detected in the blank samples, it will be reviewed to address possible procedural changes to eliminate the source of contamination. As discussed in USEPA's CLP SOW for organics analysis (USEPA 1988a) and the Functional Guidelines for Organics (USEPA 1988b), acetone, 2-butanone, methylene chloride, toluene, and the phthalate esters are considered by USEPA to be common laboratory contaminants. In accordance with the *Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II*, December 1996, if the blank contained detectable levels of common laboratory contaminants, then the sample results may be considered as positive results only if the concentrations in the sample exceeded the amount detected in the blank by one order of magnitude (10 times). For compounds other than those identified by USEPA to be common laboratory contaminants, a factor of five will be used.

#### **ACCURACY – SPIKE PERCENT RECOVERY**

Matrix spikes and surrogate spikes will be used to evaluate the accuracy of the laboratory. Both matrix spikes and surrogate spikes will be considered on the basis of percent recovery. Matrix spike percent recoveries will be calculated by the equation below.

$$\% \text{ Recovery} = \frac{\text{Result of spiked sample} - \text{Result of unspiked sample}}{\text{Amount of spike}} \times 100$$

Surrogate spike percent recoveries will be calculated by the equation below.

$$\% \text{ Recovery} = \frac{\text{Amount of surrogate detected}}{\text{Amount of surrogate added}} \times 100$$

#### **ACCURACY – OTHER**

Laboratory accuracy will also be evaluated based on QC criteria for instrument calibration, laboratory control samples, internal standards, and holding time as presented in Forms I, L, and M.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 36 of 39

## **Form R (continued)**

### **Laboratory Data Evaluation**

#### **COMPLETENESS**

Completeness is a measure of the amount of valid data obtained from the measurement system compared to the amount that is expected. Completeness will be calculated according to the following equation.

$$\text{Percent Completeness} = \frac{\text{number of valid results}}{\text{total number of results}} \times 100\%$$

Usable results will be defined for each task by addressing accuracy, precision, and representativeness parameters quantitatively. If a 90 percent or greater completeness for the analysis of VOCs in ground water samples cannot be maintained, both field techniques and laboratory techniques will be reviewed.

## **Form S**

### **Data Usability and Project Evaluation**

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated. The results of these evaluations will be included in the project report.

#### **COMPLETENESS**

For each matrix that was scheduled to be sampled, the FOL will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the GeoTrans PM determines that the deviations compromise the ability to meet project objectives (s)he will consult with the USEPA and other project team members, as necessary, to develop appropriate corrective actions.

#### **PRECISION**

The Project QA/QC Officer will determine whether precision goals for field duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Form L. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

#### **ACCURACY**

The Project QA/QC Officer will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Form M. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability (criteria in Form I); internal standard performance; and analyte recoveries for surrogates, matrix spike, matrix spike duplicate, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

#### **REPRESENTATIVENESS**

A project scientist identified by the GeoTrans PM will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and analyzed in accordance with this QAPP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

**Form S (continued)**  
**Data Usability and Project Evaluation**

**COMPARABILITY**

The Project QA/QC Officer will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless the Project QA/QC Officer indicates that such quantitative analysis is required.

**SENSITIVITY**

The Project QA/QC Officer will determine whether project sensitivity goals listed in Form K are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described in the project report.

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. The Project Manager will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration maps will be generated as part of the overall data evaluation process.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**Revision Number:** 0  
**Revision Date:** 3/25/10  
**Page:** 39 of 39

## **References**

ATSDR, 1995. Public Health Assessment Addendum, Wells G&H, Woburn, Middlesex County, Massachusetts. U.S. Department of Public Health and Human Service, Public Health Service Agency for Toxic Substance and Disease Registry. December 1995.

GeoTrans, 2010, Wells G&H Superfund Site Vapor Intrusion Assessment Work Plan, Revision 1, March 25, 2010.

The Johnson Company, 2010a, Quality Assurance Project Plan, Revision 1, Indoor Air Quality and Vapor Intrusion Assessment, UniFirst Property, Wells G&H Superfund Property, March 2010.

The Johnson Company, 2010b, Indoor Air Quality and Vapor Intrusion Assessment Work Plan, Revision 2, UniFirst Property, Wells G&H Superfund Property, March 2010.

USEPA, 2004, Second Five Year Review Report for Wells G&H Superfund Site, Woburn, Middlesex County, Massachusetts, September 30, 2004 (USEPA, 2004)

USEPA, 2010, Letter re: EPA review of UniFirst's draft VI Scope of Work, dated October 9, 2009. February 25, 2010.

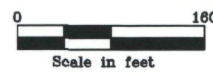


NOTE: PROPERTY BOUNDARY INFORMATION IN THE DEWEY AVE. NEIGHBORHOOD FROM MAP TITLED "TAX MAP 26 OF THE CITY OF WOBURN MASSACHUSETTS" PREPARED BY THE CITY OF WOBURN ENGINEERING DEPARTMENT




EXPLANATION

- APPROXIMATE PROPERTY BOUNDARIES DEWEY AVE. NEIGHBORHOOD
- RW10 ○ RECOVERY WELL LOCATION
- UC6 ○ MONITORING WELL LOCATION
- UG14 ● PROPOSED NEW WATER TABLE MONITORING WELL
- S81 ○ SHALLOWEST WELL AT EXISTING LOCATION PROPOSED FOR SAMPLING



TITLE:  
**MONITORING WELLS FOR VI ASSESSMENT**

LOCATION:  
**Wells G & H, Woburn, MA.**



APPROVED JRB

DRAFTED RMK

PROJECT# 117-3008048

DATE MAR 2010

FIGURE  
**1**

G:\GRACE\GENERAL\2010\01-10-VAPOR-WELLS\PRO-WELLS GRACE\UNIFIRST-SAMPLING.DWG



## APPENDIX 1

## **APPENDIX 1**

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### **Analytical Method SOPs**

**KATAHDIN ANALYTICAL SERVICES, INC.**  
**STANDARD OPERATING PROCEDURE**

**SOP Number: CA-202**  
**Revision History**  
**Cover Page**  
**Page 1**

**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: J. Halay Date: 01/20/01

Operations Manager: J. C. Burton Date: 1/15/01

QA Officer: L. Nadeau Date: 1/23/01

General Manager: D. F. Huffman Date: 1/16/01

**Revision History:**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	En	1/23/01	1/23/01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	En	5/23/01	5/23/01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	En	5/21/02	5/21/02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	Minor changes rewording of sect. 7.6.3 preservation of carbonaceous soils	LAD	02/03/05	02/03/05

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260 B	Added references, setup and operation for the Eicon / Centurion autosampler / Purge and trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practice and correct types.	LAD	04/06	04/06
09 8260 B	Sec. 4.4 added list of peak streams generated and location of peak lists. Clarified RT window studies. Added reference to MI Sop. Removed Grand mean calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying calibration verification Std. Criteria and corrective action. Reworded Correlation coefficient criteria	LAD	LAD 7-25-07 03/07 07/07	03/07 07/07
10	Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM version 4.1 criteria	LAD	08/09	08/09

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TITLE:       **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

---

I acknowledge receipt of copy \_\_\_\_ of document **SOP CA-202-10**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_ of document **SOP CA-202-10**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

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TITLE:       **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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## **1.0   SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

### **1.1   Definitions**

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

**ANALYTICAL BATCH:** 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

**METHOD BLANK** (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**CALIBRATION CHECK:** Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

**CALIBRATION STANDARD (WORKING STANDARD):** A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

**INDEPENDANT CALIBRATION STANDARD:** A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

**LABORATORY CONTROL SAMPLE (LCS):** A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD):** Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

**STANDARD CURVE (CALIBRATION CURVE):** A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

**STOCK STANDARD SOLUTION:** A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

**SURROGATES:** Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

**TARGET:** A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

**TARGET DB:** An oracle database used to store and organize all Target data files.

**QUICKFORMS:** A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Demonstration of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.



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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

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**2.0 SUMMARY OF METHOD**

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

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**3.0 INTERFERENCES**

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire Tekmar or Archon must be backflushed with warm methanol and water.

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**4.0 APPARATUS AND MATERIALS**

- 4.1 GC: Hewlett Packard 6890 & 5890
- 4.2 Mass Spectrometers (MS): HP5973, HP5972 and HP5970

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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- 4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.
- 4.4 Columns: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.
- 4.5 Purge and Traps: Archon 5100, Tekmar 2016 and Centurion auto samplers, and Tekmar 2000, 3000 and Encon concentrators.
- 4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.
- 4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.
- 4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.
- 4.9 Data System: The Target software is used for processing data and generating forms.

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**5.0 REAGENTS**

- 5.1 Purge and trap grade methanol
- 5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.
- 5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".
  - 5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.
  - 5.3.2 Secondary dilution standards

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Acetone	Dibromochloromethane	P-Isopropyltoluene
Benzene	1,2-Dibromoethane	Methylene Chloride
Bromobenzene	Dibromomethane	4-Methyl-2-Pentanone
Bromochloromethane	1,2-Dichlorobenzene	Naphthalene
Bromodichloromethane	1,3-Dichlorobenzene	N-Propylbenzene
Bromoform	1,4-Dichlorobenzene	Styrene
Bromomethane	Dichlorodifluoromethane	1,1,1,2-Tetrachloroethane
2-Butanone	1,1-Dichloroethane	1,1,2,2-Tetrachloroethane
n-Butylbenzene	1,2-Dichloroethane	Tetrachloroethene
sec-Butylbenzene	1,1-Dichloroethene	Tetrahydrofuran
tert-Butylbenzene	cis-1,2-Dichloroethene	Toluene
Carbon Disulfide	Trans-1,2-Dichloroethene	1,2,3-Trichlorobenzene
Carbon Tetrachloride	1,2-Dichloropropane	1,2,4-Trichlorobenzene
Chlorobenzene	1,3-Dichloropropane	1,1,1-Trichloroethane
Chloroethane	2,2-Dichloropropane	1,1,2-Trichloroethane
2-Chloroethylvinyl Ether	1,1-Dichloropropene	Trichloroethene
Chloroform	Cis-1,3-Dichloropropene	Trichlorofluoromethane
Chloromethane	Trans-1,3-Dichloropropene	1,2,3-Trichloropropane
2-Chlorotoluene	Ethylbenzene	1,2,4-Trimethylbenzene
4-Chlorotoluene	Hexachlorobutadiene	Vinyl Acetate
Cyclohexane	2-Hexanone	Vinyl Chloride
1,2-Dibromo-3-Chloropropane	Idomethane	1,3,5-Trimethylbenzene
Isopropylbenzene	Methyl Tert-Butyl Ether	1-Chlorohexane

5.3.2.2 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 200 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl Alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl Chloride	Methyl Acetate
Chloroprene	Methyl Methacrylate
Diethyl Ether	Methyl Tert-Butyl Ether
Cis-1,4-Dichloro-2-Butene	Pentachloroethane
Trans-1,4-Dichloro-2-Butene	Propionitrile
1,4-Dioxane	Tertiary-Amyl Methyl Ether
Di-Isopropyl Ether	Tertiary-Butyl Alcohol
Ethyl Methacrylate	1,3,5-Trichlorobenzene
Ethyl Tertiary-Butyl Ether	1,2,3-Trimethylbenzene
Freon-113	

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TITLE:           **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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5.3.2.3 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

5.3.2.4 Surrogate Spiking Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

4-Bromofluorobenzene  
1,2-Dichloroethane-D<sub>4</sub>  
Toluene-D<sub>8</sub>  
Dibromofluoromethane

5.3.2.5 Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Pentafluorobenzene  
1,4-Difluorobenzene  
Chlorobenzene-D<sub>5</sub>  
1,4-Dichlorobenzene-D<sub>4</sub>

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

5.3.2.7 See Table 4 for a complete list of standards, concentration, and vendors.

**NOTE:** The concentrations of standards may vary depending on the type of autosampler being used.

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TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

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## 6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved. All soil/sediments must be analyzed within 14 days from sample collection. For specific projects, soil may be received in pre-weighed vials containing methanol, with an aliquot of the methanol used for analysis. For these projects, the methanol aliquot must be analyzed within 14 days from sample collection. Samples must be stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  from the time of receipt at the lab until analysis.

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## 7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files: I826AXX.M (all samples and standards)

Where:

XX = the calibration number in chronological order

I = instrument ID (F,M,S,T, or Z)

A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

BFB288AQ.M (waters) or BFB288SL.M (soils) (BFB tuning acquisition)

Data files for BFB: IB\_\_\_.D where \_\_\_ is a number in chronological order from 000 to 999, and I is the instrument ID (F,M,S,T, or Z).

All other data files: I\_\_\_\_.D where \_\_\_\_ is a number in chronological order from 0000 to 9999, and I is the instrument ID (F,M,S,T, or Z). This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

<u>Mass</u>	<u>Criteria</u>
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

Column:	RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures: Injection port:	200°
Transfer line:	150°
Detector:	240°
Isothermal temperature:	150°
Run time:	8 minutes
Scan start time:	3 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts



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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

#### 7.4 INSTRUMENT CONFIGURATION / CALIBRATION

7.4.1 Tekmar LSC 3000/Archon 5100/ Tekmar 2016, Setup/Operation: Please refer to the Tekmar or Archon Manuals for more detailed operations for these instruments.

To begin, set the Tekmar LSC 2000/3000 to the specification listed in section 2-12 of the Archon manual. Edit method 14 as follows:

Method 14 should include:

Standby:	35°
Prepurge:	0 min
Preheat Temp:	0°
Sample Temp:	0°
Purge:	11 min
Dry purge:	2-4 min
Desorb preheat:	245°
Desorb Temp:	250°
Desorb time:	2-5 min
Dry purge:	2-4 min
Bake Time:	10 min
Bake Temp:	260°
Auto drain:	On
Bake gas by pass:	Off
Valve Temp:	120°
Line Temp:	120°
Runs per sample:	1

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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The above temperature settings are for a Vocab 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon; the settings for this feature will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

#### 7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:      Purge Gas: Helium  
                                Purge Time: 11.0 ± 0.1 minute  
                                Purge Flow Rate: approx. 24-40 mL/min  
                                Purge Temperature: Ambient (water)

Desorb Conditions:      DesorbTemp: 250°C  
                                Desorb Flow rate: 15 mL/min  
                                Desorb Time: 2.0 ± 0.1 min  
                                Bake Time: 10 min  
                                Bake Temperature: 260°C

The above temperature settings are for a Vocab 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

#### 7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 200 ug/L. The calibration standards are stirred and heated to 40°C.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

	STD. ID	CAL. Mix 200 ug/mL	Extras Mix 200 ug/mL	Surr. Mix 250 ug/mL Archon	Surr. Mix 50 ug/mL Centurion
AQ curve only	VSTD001	0.5 uL	0.5 uL	0.4 uL	2.0 uL
	VSTD005	2.5 uL	2.5 uL	2.0 uL	10 uL
SL curve only	VSTD010	5.0 uL	5.0 uL	4.0 uL	20 uL
	VSTD020	10 uL	10 uL	8.0 uL	40 uL
CC	VSTD050	25 uL	25 uL	20 uL	100 uL
	VSTD100	50 uL	50 uL	40 uL	200 uL
	VSTD200	100 uL	100 uL	80 uL	400 uL

The internal standard is spiked by the autosampler. Due to different spike amounts separate standards are used depending on which autosampler is being used.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

#### 7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

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TITLE:           **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

#### 7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient ( $r$ ) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it ( $r^2$ ). This is what is reported on all Target forms. The value for  $r^2$  must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

#### 7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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For projects or clients requiring DoD QSM 4.1 all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

#### 7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.3, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM 4.1 all project analytes must have  $\pm 20\%D$ .

Continuing calibration check criteria must be met before sample analysis can proceed.

#### 7.4.7 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

For projects or clients requiring DoD QSM 4.1, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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**7.5 QUALITY CONTROL SAMPLE ANALYSIS**

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

**7.5.1 Laboratory Control Sample (LCS)**

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25  $\mu$ L of the LCS standard mix at 200  $\mu$ g/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50  $\mu$ g/L. The Archon autosampler adds 1  $\mu$ L of internal and 1  $\mu$ L of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5  $\mu$ L of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

To prepare the water and medium-level soil LCS for analysis on the LSC 2000 / 2016 autosampler, 1.25  $\mu$ L of the LCS standard mix at 200  $\mu$ g/mL are spiked into 5 mL of analyte-free laboratory reagent grade water for a final concentration of 50  $\mu$ g/L.

**7.5.2 Method Blank Analysis**

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.



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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM 4.1 no analytes may be detected  $>1/2$  the PQL and  $>$  than the  $1/10^{\text{th}}$  the measured amount in any sample or  $1/10^{\text{th}}$  the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected  $>$  than the PQL.

#### 7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

#### 7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two ( $-50\%$  to  $+100\%$ ), from the last daily calibration standard, the GC/MS must be inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

#### 7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

### 7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, "Basic Laboratory Technique", current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

#### 7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

##### 7.6.1.1 Tekmar LSC 2000 / 2016 units

Rinse a 5.0 mL gas-tight syringe a minimum of three times with analyte-free laboratory reagent grade water (e.g., Poland Spring or equivalent). Pour sample at ambient temperature into the syringe until nearly overflowing. Carefully insert and adjust plunger to sample volume of 5.0 mL. While adjusting plunger to final volume, expel extra volume of sample onto pH paper for sample pH verification. Add 1.0 uL of the internal and surrogate mixtures (250 ug/mL). Immediately inject contents of the syringe into the ALS sparger.

Record the sample pH in the injection logbook. Continue as above for each sample, ensuring that the 5.0 mL gas-tight syringe is rinsed a minimum of three times with laboratory reagent grade water between each sample.

##### 7.6.1.2 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

##### 7.6.1.3 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor both the samples immediately after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

#### **7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES**

Method 5035 Closed System Purge & Trap procedure for low level soils  
(5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the Tekmar or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. This means that the Tekmar ALS 2016 unit may be used for the preparative step, as well as the Archon units.

### 7.6.3 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

**Bulk Sample-** A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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### Extraction

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

## 7.7 FINAL DATA PACKAGE

### 7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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elements, the chromatography and quantitation of target analytes must be reviewed.

#### 7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

#### 7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within  $\pm 20\%$  between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.



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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

#### 7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

#### 7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

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### 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

#### 8.1 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

For projects or clients requiring DoD QSM 4.1 all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

## **8.2 Surrogate Recovery Criteria**

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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### 8.3 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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## 9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

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## 10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 8260B, current revision.

"Department of Defense Quality Systems Manual for Environmental Laboratories" (DoD QSM), Version 4.1, 04/22/09.

"The National Environmental Laboratory Accreditation Conference (NELAC) Standards," June 2003.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

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**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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**LIST OF TABLES AND FIGURES**

TABLE 1	QC REQUIREMENTS
TABLE 2	SUMMARY OF METHOD MODIFICATIONS
TABLE 3	VOA COMPOUNDS & CHARACTERISTIC IONS
TABLE 4	ANALYTE QUANTITATION AND INTERNAL STANDARDS
FIGURE 1	EXAMPLE OF VOA RUNLOG PAGE
FIGURE 2	EXAMPLE OF STANDARDS RECEIPT LOG
FIGURE 3	EXAMPLE OF STANDARDS PREP LOG
FIGURE 4	STANDARD INFORMATION

**TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

**TABLE 1**

**QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260**

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq 0.30$ , except chloromethane, 1,1-DCA and bromoform $\geq 0.10$ ; RSD for RFs $\leq 30\%$ for CCCs. Refer to section 7.4.3 also.	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances  For projects requiring DoD QSM 4.1, all target analytes must have $\pm 20\%$ recovery of true value.	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are $<PQL$ , narrate. Otherwise, reprep a blank and the remaining samples.  For projects requiring DoD QSM 4.1, no samples may be run until a passing ICV is run.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF $\geq 0.30$ , except chloromethane, 1,1-DCA and bromoform $\geq 0.10$ ; RF for CCC analytes $\leq 20\%$ (%D) of average initial multipoint RF  For projects requiring DoD QSM 4.1, the %Difference/Drift for all target analytes must be less than or equal to 20%	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time $\pm 30$ seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS  For projects requiring DoD QSM 4.1, IS responses and retention times are compared to the midpoint of the most recent ICAL for all samples and QC.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning



TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method Blank	One per batch of 20 or fewer samples.  For projects requiring DoD QSM 4.1, one per preparatory batch.	No analytes of interest detected > PQL with the exception of Methylene Chloride  See section 7.5.2 of this SOP for additional DoD acceptance requirements.	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.  For projects requiring DoD QSM 4.1, DoD limits shall be used, unless otherwise specified by the project QAPP.	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.  For projects requiring DoD QSM 4.1, DoD limits shall be used, if available. Otherwise lab limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.  For projects requiring DoD QSM 4.1, one MS/MSD pair will be analyzed per preparatory batch per matrix if supplied with sufficient sample.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.  For projects requiring DoD QSM 4.1, DoD limits shall be used, unless otherwise specified by the project QAPP.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2  
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-10	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl Ether	45	43, 87
Ethylbenzene	91	106
Ethyl Methacrylate	69	41, 99
Ethyl Tertiary-Butyl Ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl Alcohol	43	41, 42
Isopropylbenzene	105	120
P-ISOPROPYLTOLUENE	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene Chloride	84	86, 49
Methyl Acetate	43	74
Methyl Methacrylate	69	41, 100
4-Methyl-2-Pentanone	43	58, 85, 100
Methyl Tert-Butyl Ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
N-PROPYLBENZENE	91	120
Styrene	104	78
Tertiary-Amyl Methyl Ether	73	55, 87, 71
Tertiary-Butyl Alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132

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TITLE:           **ANALYSIS OF VOA<sub>s</sub> BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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TABLE 3 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Acetate	43	86
Vinyl Chloride	62	64
Xylenes (Total)	106	91
1-Chlorohexane	91	55,43

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenze	1,4-Difluorobenzene	Chlorobenzene - d5	1,4-Dichlorobenzene - d4
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butylether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-aryl methyl ether			Pentachloroethane
Diethyl Ether			n-Butylbenzene
Carbon Disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
1-Chlorohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			



TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES  
GCMS-T INSTRUMENT RUNLOG







DATE/TIME OF BFB INJECTION: 8/6/09 08:52  
Reviewed by/Date:

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD			Y/N	MS/MSD	PH	ANALYST	COMMENTS
					5030	5035	1311					
SO N/A BFB	TR906	-	-	BFA288A0				Y			HCG	
VSTN55706A	TSS33	1	1	T826A31				Y				
LCSA W6167144-1	TSS34	1	2					Y				
VKA W6167144-1	TSS35	1	3					N				
↓ B W6167144-2	TSS36	1	4					Y				target wide
SL4258-1	A TSS37	1	5		X			Y	1	22		
-6	B TSS38	1	6					Y	2	22		
SL4351-1	A TSS39	1	7					Y	3	22		
-6	A TSS40	1	8					Y	4	22		
SL4337-7	A TSS41	1	9					Y	5	22		
SL4258-2	B TSS42	1	10					Y	6	22		
-4	B TSS43	1	11					Y	7	22		
SL4351-2	B TSS44	1	12					N	8	22		1561 RR
-4	B TSS45	1	13					Y	9	22		
-7	B TSS46	1	14					Y	10	22		
SL4337-1	A TSS47	1	15					Y	11	22		
-2	A TSS48	1	16					Y	12	22		
-3	A TSS49	1	17					Y	13	22		
-4	C TSS50	1	18					Y	14	22		
5-6	C TSS51	1	19					Y	15	22		
3019 5-5	E TSS52	1	20					Y	16	22		
SL4472-1	A TSS53	1	21					Y	17	22		
-2	A TSS54	1	22		↓			Y	18	22		20:29 ✓
RINSE	TSS55	2	23					-	-	-		
↓	TSS56	2	24	↓				-	-	-	↓	
8/7/09												
STANDARD	CODE	STANDARD		CODE	Circle Methods:							
BFB	V2802	IS MIX	V2810	SW846 8260	OLM 04.2							
CAL STD.	V2808	SS MIX	V2811	EPA 624	OLM 03.1							
LCS/MS MIX	V2809			EPA 524	OLC 02.1							
EXTRAS MIX	V2797			SIM	OLC 03.2							

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES			
STOCK STANDARDS RECEIVED			
GC/MS LABORATORY REVIEWED BY/DATE:			
AMP1514 1519 1520 1521	1-Chlorohexane (EPA-1208) Lot: CB-8341A Ultra Exp: 11/30/09 1000µg/mL		Rec'd 11/07/07
AMP1522 1523 1524 1525	 Cat# 80434 HA VPI Standard 600-1600 µg/mL in Purge and Trap Methanol Lot # A040187 Exp: 11/12 Store: Freezer Restek Corporation - 110 Bowers Circle - Bellefonte, PA 16823		Rec'd 11/7/07 FKL ↓
AMP1526 1527	 Cat# 80465 Galkon Reproduction #1 200-1000 µg/mL in F11 Standard Lot # A049282 Exp: 12/11 Store: Freezer Restek Corporation - 110 Bowers Circle - Bellefonte, PA 16823	RL FOR LABORATORY USE ONLY	rec'd DMF 11/20/07
AMP1528 1529	 AS-E0265 Diethyl ether 5000 µg/mL in MeOH Lot: B-070099-1A Exp: Jan 19, 2010	1 mL STORAGE Ambient POISON	rec'd DMF 11/21/07 ↓
AMP1530 1531	 CLP-LC-1S-100X Laboratory Control Sample - Internal Standard Mix 2500 µg/mL in MeOH Lot: B2090027 Exp: Sep 6, 2012	1 mL 3 comps. STORAGE Ambient POISON	↓
AMP1532 1533	 Cat# 80624 G18 H1 H8 G182 Non-halogenated Single peak standard Lot # A050631 Exp: 3/10 Store: Freezer Restek Corporation - 110 Bowers Circle - Bellefonte, PA 16823	RL FOR LABORATORY USE ONLY	rec'd DMF 11/26/07

TITLE:

### EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE

(HGW) 35109

815709

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 4

STANDARD INFORMATION

VOA Standards

Standard	Concentration	Manufacturer	Catalog Number
1,2,3 Trimethylbenzene	2000 ug/mL	Restek	56733
1,2,3 Trichlorobenzene	2000 ug/mL	Accustandard	M-502-47-10X
1,2,4 Trimethylbenzene	2000 ug/mL	Accustandard	M-502-54-10X
1,3,5 Trichlorobenzene	neat	Supelco	44-2235
1,3,5 Trimethylbenzene	2000 ug/mL	Accustandard	M502-55-10X
2-CEVE	2000 ug/mL	Accustandard	M-601C-10X
502.2 Cal Mix #1 (gases)	2000 ug/mL	Restek	30042
502.2 Cal2000 Mega Mix	2000 ug/mL	Restek	30431
504.1 Cal Mix	200 ug/mL	Accustandard	M-504.1-CSS
Acrolein & Acrylonitrile	5000 ug/mL	Accustandard	M-603-M-5X
Appendix IX Volatiles Mix	various	Accustandard	M-8240C-R3-10X
Bromochloromethane	2000 ug/mL	Accustandard	M-502-03-10X
California Oxygenates Mix #1	2000 - 10,000 ug/mL	Restek	30465
Carbon Disulfide	2000 ug/mL	Restek	30258
Chloroprene	2000 ug/mL	Accustandard	APPX9-048-R1
Custom GC Std	2000 ug/mL	Accustandard	S-11160
Custom VOC mix	various	Accustandard	S-7920-R1
Custom Volatile GC/MS Std	2000 ug/mL	Accustandard	S-3432B
Custom Volatiles GC/MS	2000 ug/mL	Accustandard	S-3432A
Dietheyl Ether	5000 ug/mL	Accustandard	AS E0285
Freon 113	2000 ug/mL	Supelco	4-7944
Method 8260 Additions	2000 ug/mL	Accustandard	M-8260-ADD-10X
Method 8260B-Revision	2000 ug/mL	Accustandard	M-8240B-R-10X
MTBE	2000 ug/mL	Supelco	4-8483
Napthalene	2000 ug/mL	Accustandard	M-502-40-10X
THF	2000 ug/mL	Accustandard	S-4575-10X
Vinyl Acetate	2000 ug/mL	Restek	30216
Vinyl Acetate	2000 ug/mL	Accustandard	APPX9-211-20X
VOA Calibration Mix #1 (Ketones)	5000 ug/mL	Restek	30006
TCL Ketone Mix	5000 ug/mL	Accustandard	CLP-022-25X
VOC Liquid Mix	2000 ug/mL	Accustandard	M-502A-R2-10X
Volatile Organic Compounds (gases)	2000 ug/mL	Accustandard	M-502B-10X
<b>IS/SS/Tune</b>			
Custom 8260 IS	5000 ug/mL	Restek	54577
Custom 8260 SS	5000 ug/mL	Restek	54578
4-BFB	2000 ug/mL	Supelco	48083
VOA Tuning Compound (BFB)	5000 ug/mL	Restek	30003
1,2 Dichlorobenzene-D4	2000 ug/mL	Supelco	48952-U
Fluorobenzene	2000 ug/mL	Supelco	
VOA IS (CLP)	2500 ug/mL	Restek	30004
VOA SS (CLP)	2000 ug/mL	Supelco	48943
624 IS	1500 ug/mL	Restek	30023
4-BFB/Fluorobenzene/Pentafl. (EPA 624)	20000 ug/mL	Accustandard	M-624-SS-M
8260A SS	2500 ug/mL	Restek	30240
<b>CLP Only</b>			
04.1 CLP VOA Cal 2000	2000 ug/mL	Restek	30456
LCS-IS	2500 ug/mL	Accustandard	CLP-LCS-IS-100X
LCS-Volatiles	200 ug/mL	Accustandard	CLP-LCS-V
CLP Volatiles DMC Stock Solution	deutrated compds	Cambridge Isotope	ES 5038
3.2 OLC mix	1000 - 2000 ug/mL	Restek	30492

**KATAHDIN ANALYTICAL SERVICES, INC.**  
**STANDARD OPERATING PROCEDURE**

**SOP Number: CA-220**  
**Revision History**  
**Cover Page**  
**Page 1**

**TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:**  
**SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

Prepared By: GC/MS Group Date: 07/01

Approved By:

Group Supervisor: J. Haly Date: 080307

Operations Manager: John C. Burton Date: 08/03/01

QA Officer: Deborah J. Nadeau Date: 8/6/01

General Manager: Deborah F. Kufra Date: 8/6/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8260B	Minor change to criteria in section 7.5.4. Updated calibration standard mixes.	En	5/23/02	5/23/02
02 8260B	Reorganization of sections 4, 5, 6 and 7 and Tables and Figures. Added definitions and information for the new data processing system.	MRC	01.06.04	01.06.04
03 8260B	Corrected %RSD for Initial Cal. in Sect. 7.4.3 & Table 1 added wording to Sects 6 and 8 minor changes throughout	LAD	020405	020405
04 8260B	Change Title to include all VOCs. Added instrumentation to sect. 4.4, 7.4.2 & 7.6.3 update std. conc. sects 5.3.2.1, 5.3.2.2, 5.3.2.3, 7.4.4, 7.5.1 & 7.5.9 Added "T" instr. and removed "Q" instr. Updated cal. information. added compounds to Table 3. updated IS and SS mixes	LAD	04/06	04/06
05	Sect 4.7 - changed syringe sizes Sect. 5.2 - changed milli Q to Semans Sect. 7.3 - changed run time Sect. 7.4.1 - changed desorb time Added ICV criteria, MI references and RT window criteria.	LAD	06/07	06/07

**SOP Number: CA-220**  
**Revision History**  
**Cover Page Cont.**  
**Page 2**

**TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

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KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

SOP Number: CA-220-07  
Date Issued: 08/09  
Page 3 of 32

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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**TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

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## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze aqueous samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision, modified for selected ion monitoring to achieve lower detection levels for Volatile Organic Compounds (VOCs).

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

### **1.1 Definitions**

VOC Volatile Organic Compounds

VOA Volatile Organic Analysis

**ANALYTICAL BATCH:** 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

**METHOD BLANK (laboratory reagent blank):** A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**CALIBRATION CHECK:** Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

**CALIBRATION STANDARD (WORKING STANDARD):** A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

**LABORATORY CONTROL SAMPLE (LCS):** A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination. When analyzed directly after a calibration, the LCS doubles as the Independent Calibration Verification (ICV).

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD):** Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to

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**TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

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sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

**STANDARD CURVE (CALIBRATION CURVE):** A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

**STOCK STANDARD SOLUTION:** A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

**SURROGATES:** Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

**TARGET:** A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

**TARGET DB:** An oracle database used to store and organize all Target data files.

**QUICKFORMS:** A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and USEPA CLP report forms and disk deliverables, which can be customized.

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260 modified for SIM. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatiles organics by Method 8260, modified for SIM, to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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## 2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. These volatile organics are then retained on a cooled trap (SP1000/tenax/silica gel medium, or equivalent) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

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## 3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. When a Tekmar ALS is used, the contaminated position cannot be used until a clean blank can be obtained from that position. A clean blank is a blank for which any analytes of interest are not detected at concentrations greater than the laboratory PQLs. If the lines are determined to be contaminated, then the entire Tekmar must be backflushed with warm methanol and water.

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## 4.0 APPARATUS AND MATERIALS

- 4.1 GC: Hewlett Packard 5890s and 6890
- 4.2 Mass Spectrometers (MS): HP5972s and HP5973
- 4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber components are not to be used.
- 4.4 Columns: DB-624, 25 meter, 0.2mm ID or RTX-VMS, 40 meter, 0.18mm ID
- 4.5 Purge and Traps: Archon 5100, Tekmar 2016 and Centurion auto samplers, and Tekmar 2000, 3000 and Encon concentrators.
- 4.6 Purge tube: 25 ml fritted purge vessel.
- 4.7 Hamilton Gastight syringes: 5.00 uL to 25.00 mL.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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- 4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.
- 4.9 Data System: The Target software is used for processing data and generating forms.

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## 5.0 REAGENTS

- 5.1 Purge and trap grade methanol
- 5.2 Organic-free laboratory reagent grade water: Siemens Water Technologies. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.
- 5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".

- 5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

- 5.3.2 Secondary dilution standards

- 5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the components listed below. The final concentration of each component is 5.0 ug/mL (25 ug/mL for 4-methyl-2-pentanone). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Chloromethane	Trichloroethene	1,2-Dibromomethane
Vinyl Chloride	Bromodichloromethane	1,1,1,2-Tetrachloroethane
Trichlorofluoromethane	cis-1,3-Dichloropropene	1,1,2,2-Tetrachloroethane
1,1-Dichloroethene	trans-1,3-Dichloropropene	1,2,3-Trichloropropane
Chloroform	4-Methyl-2-pentanone	1,4-Dichlorobenzene
Carbon Tetrachloride	Tetrachloroethene	1,2-Dibromo-3-chloropropane
Benzene	Dibromochloromethane	Hexachlorobutadiene
1,2-Dichloroethane	1,3-Dichloropropene	

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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5.3.2.2 Laboratory Control Spike and MS/MSD Mixture - Prepare a standard independent from the calibration standards, as above containing the compounds listed below. The final concentration of each component is 5.0 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Chloromethane	Trichloroethene	1,2-Dibromomethane
Vinyl Chloride	Bromodichloromethane	1,1,1,2-Tetrachloroethane
Trichlorofluoromethane	cis-1,3-Dichloropropene	1,1,2,2-Tetrachloroethane
1,1-Dichloroethene	trans-1,3-Dichloropropene	1,2,3-Trichloropropane
Chloroform	4-Methyl-2-pentanone	1,4-Dichlorobenzene
Carbon Tetrachloride	Tetrachloroethene	1,2-Dibromo-3-chloropropane
Benzene	Dibromochloromethane	Hexachlorobutadiene
1,2-Dichloroethane	1,3-Dichloropropene	

5.3.2.3 Surrogate Spiking Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each component is 6.25 ug/mL. The standard must be prepared every 14 days and stored on the Archon in a pressurized vial or in the VOA standards freezer between uses.

DBFM
1,2-Dichloroethane-d4
Toluene-d8
p-Bromofluorobenzene

5.3.2.4 Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each component is 25 ug/mL. The standard must be prepared every 14 days and stored on the Archon in a pressurized vial or in the VOA standards freezer between uses.

Pentafluorobenzene
1,4-Difluorobenzene
Chlorobenzene-d5
1,4-Dichlorobenzene-d4

5.3.2.5 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

5.3.2.6 See Table 5 for a complete list of standards, concentration, and vendors.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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## 6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are sampled into 40 mL glass VOA vials leaving no headspace. Samples are preserved with 2 drops 1:1 HCL and stored at 4°C ( $\pm 2^{\circ}\text{C}$ ) until analysis.

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved.

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## 7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files: YSIMAXX.M (all samples and standards)

Where:

XX = the calibration number in chronological order

Y = instrument ID (D,F,M,S,T, or Z)

BFB288AQ.M (BFB tuning acquisition)

Data files for BFB: IB\_\_\_.D where \_\_\_ is a number in chronological order from 000 to 999, and I is the instrument ID (D, F,M,S,T, or Z).

All other data files: I\_\_\_\_.D where \_\_\_\_ is a number in chronological order from 0000 to 9999, and I is the instrument ID (D, F,M,S,T, or Z). This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:



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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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<u>Mass</u>	<u>Criteria</u>
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

7.3.1.2 **GC/MS type: 5972s and 5973**

Column:	DB-624, 25 meter, 0.2 mm I.D. or RTX-VMS, 40meter, 0.18mmID.
Temperatures:	Injection port: 200° Transfer line: 150° Detector: 240°
Isothermal temperature:	150°
Run time:	6-8 minutes
Scan start time:	3 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 8260 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your department manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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#### 7.4 INSTRUMENT CONFIGURATION / CALIBRATION

- 7.4.1 Tekmar LSC 3000/Archon 5100/ Tekmar 2016, Setup/Operation: Please refer to the Tekmar or Archon Manuals for more detailed operations for these instruments.

To begin, set the Tekmar LSC 2000/3000 to the specification listed in section 2-12 of the Archon manual. Edit method 14 as follows:

Method 14 should contain:

Stand by 35°	Prepurge 0 min
Preheat 0°	Sample 0°
Purge 11 min	Dry purge 2-4 min
Desorb preheat 245°	
Desorb 250°	Desorb time 2 min
Bake 10 min	Bake 260°
Auto drain	On
Bake gas by pass	Off
Valve 120°	Line 120°
Runs per sample	1

The above temperature setting are for a Vocab 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon, the settings for these feature will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

#### 7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:	Purge Gas: Helium
	Purge Time: 11.0 ±0.1 minute
	Purge Flow Rate: approx. 24-40 mL/min
	Purge Temperature: Ambient (water)

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**TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)**

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Desorb Conditions: DesorbTemp: 250°C  
Desorb Flow rate: 15mL/min  
Desorb Time: 2.0 ± 0.1 min  
Bake Time: 10 min  
Bake Temperature: 260° C

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

**7.4.3 Initial Calibration for Method SIM 8260**

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

The GC/MS must be initially calibrated at six different levels (target compounds at 0.05, 0.075, 0.10, 0.25, 0.50, and 1.0 ug/L) to determine the linearity of response. See Section 5.3.2 for preparation of the calibration standards. Tables 3 and 4 contain a list of target compounds, internal standards, and surrogates with their defined primary quantitation ions.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

	Cal mix @ 5.0/25 ug/mL	SS @6.25 ug/mL
VSTD0.05	1.0 uL	0.8 uL
VSTD0.075	1.5 uL	1.2 uL
VSTD0.10	2.0 uL	1.6 uL
VSTD0.25	5 uL	4 uL
VSTD0.50	10 uL	8 uL
VSTD1.00	20 uL	16 uL

The internal standard is spiked by the autosampler.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 4.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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7.4.4. Initial Calibration Criteria

Refer to Table 1, QC Requirements, for specific criteria that must be met for Method 8260. The percent (%) RSD for VOCs must be less than or equal to 15%.

For projects or clients requiring DoD QSM 4.1 an independent calibration verification (ICV) sample must be run. The ICV must contain compounds from a different source than the ICAL. All project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met.

7.4.5. Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing the target compounds, internal standards and surrogates at the concentrations below must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB.

Standard	Conc.
VOC Mix	0.25 ug/mL
SS Mix	0.25 ug/mL
IS Mix	1.0 ug/mL

The relative response factor from the 0.25 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extended ion current profile) area for the internal standard in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for the internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for VOCs is less than or equal to 15%, the continuing calibration is considered valid.

Continuing calibration check criteria must be met before sample analysis can proceed. The VSTD0.25 analyzed as part of the initial calibration curve can be used as the continuing calibration standard, assuming all criteria are met and time is left in the twelve-hour window to analyze samples.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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#### 7.4.6. Retention Time Windows

Retention time windows for the internal standards are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows for the internal standards of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The internal standard of the samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

For projects or clients requiring DoD QSM 4.1, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

#### 7.5 Quality Control Sample Analysis

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

##### 7.5.1. Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the LCS, 5  $\mu$ L of the LCS standard mix at 5.0  $\mu$ g/mL is spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 0.25  $\mu$ g/L. The autosampler adds the internal and surrogate standard to a 25mL aliquot of this preparation for analysis. The concentration of the SS/IS mix is dependant of which autosampler is being used.

##### 7.5.2. Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM 4.1 no analytes may be detected  $>1/2$  the PQL and  $>$  than the  $1/10^{\text{th}}$  the measured amount in any sample or  $1/10^{\text{th}}$  the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected  $>$  than the PQL.

#### 7.5.3. Surrogate Recovery Limits

For blanks, laboratory control samples, and client samples, the nominal limits must be met for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

#### 7.5.4. Internal Standard Area Recoveries / Retention Times.

The internal standard response and retention time in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extended ion current profile) area for the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must be inspected, and corrective action taken. If the retention time for the internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

#### 7.5.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 25 mL aliquots (aqueous), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 0.25 ug/mL. Acceptance criteria for MS/MSD pairs are outlined in Section 8.0.

### 7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

---

about the sample. Sample history is used to determine what order in which to run the samples and at what dilution.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

#### 7.6.1 Tekmar LSC 2000 / 2016 units

Rinse a 25.0 mL gas-tight syringe a minimum of three times with analyte-free laboratory reagent grade water (e.g., Poland Spring or equivalent). Pour sample at ambient temperature into the syringe until nearly overflowing. Carefully insert and adjust plunger to sample volume of 25.0 mL. While adjusting plunger to final volume, expel extra volume of sample onto pH paper for sample pH verification. Add 1.0 uL of the internal and surrogate mixtures. Immediately inject contents of the syringe into the ALS sparger. To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: Analyze a method blank immediately after the contaminated sample and a method blank in the same purge inlet during its next use; both must not contain the target in question at a level >PQL. Monitor both the samples immediately after the contaminated sample and the sample next run in the same purge inlet for the target(s) in question; both must have levels <PQL. Continue as above for each sample, ensuring that the 25.0 mL gas-tight syringe is rinsed a minimum of three times with analyte-free laboratory reagent grade water between each sample.

#### 7.6.2 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

#### 7.6.3 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

---

Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

**For All Units:**

Record the sample pH in the injection logbook.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

## 7.7 FINAL DATA PACKAGE

### 7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.



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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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#### 7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or internal standard area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. A "m" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the department manager or his/her designee, who will review each manual integration.

For specific Manual Integration procedures, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

#### 7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within  $\pm 20\%$  between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

---

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form I as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the GC/MS Department Manager, or his designee.

#### 7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics Department is then processed by the Data Management department.

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### 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

#### 8.1 LCS Criteria

Nominal limits of 70-130% are used. Where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For projects or clients requiring DoD QSM 4.1, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used. Laboratory limits may not be greater than  $\pm 3$  standard deviations from the mean recovery.

#### 8.2 MS/MSD Criteria

Nominal limits of 70-130% are used. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For projects or clients requiring DoD QSM 4.1, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used. Laboratory limits may not be greater than  $\pm 3$  standard deviations from the mean recovery. A MS/MSD must be analyzed with each analytical/preparatory batch per matrix. RPD must be  $\leq 30\%$  between the MS and MSD.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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### 8.3 Surrogate Recovery Criteria

Surrogate Limits: (Nominal Limits) 70-130%

For projects or clients requiring DoD QSM 4.1, DoD recovery limits shall be used, when available. If DoD limits are not available laboratory limits may be used.

### 8.4 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The department manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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## 9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit and Reporting Limit Studies and Verifications for procedures on determining the MDL.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

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## 10.0 APPLICABLE DOCUMENTS/REFERENCES

"Department of Defense Quality Systems Manual for Environmental Laboratories" (DoD QSM), Version 4.1, 04/22/09.

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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"The National Environmental Laboratory Accreditation Conference (NELAC) Standards,"  
June 2003.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third  
Edition, Final Update III, December 1996, Method 8260B, current revision.

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#### LIST OF TABLES AND FIGURES

TABLE 1	QC REQUIREMENTS
TABLE 2	SUMMARY OF METHOD MODIFICATIONS
TABLE 3	CHARACTERISTIC IONS
TABLE 4	INTERNAL STANDARD
TABLE 5	STANDARD INFORMATION
FIGURE 1	EXAMPLE OF VOA RUNLOG PAGE
FIGURE 2	EXAMPLE OF STANDARDS RECEIPT LOG
FIGURE 3	EXAMPLE OF STANDARDS PREP LOG

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration	Initial calibration prior to sample analysis	RSD <15%	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration.	For projects requiring DoD QSM 4.1, all target analytes must have $\pm 20\%$ recovery of true value.	For projects requiring DoD QSM 4.1, no samples may be run until a passing ICV is run.
Calibration verification	Once per each 12 hours, prior to sample analysis	RF within 15% of average initial multi-point RF.	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	Immediately after or during data acquisition of calibration check standard	Retention time $\pm 30$ seconds; EICP area within - 50% to +100% of last calibration verification (12 hours) for IS.  For projects requiring DoD QSM 4.1, IS responses and retention times are compared to the midpoint of the most recent ICAL for all samples and QC.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples  For projects requiring DoD QSM 4.1, one per preparatory batch.	No analytes of interest detected > PQL  See section 7.5.2 of this SOP for additional DoD acceptance requirements.	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Nominal 70-130% recovery  For projects requiring DoD QSM 4.1, DoD limits shall be used, if available. Otherwise lab limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out re-extract and analyze sample (4) If reanalysis is out, flag data

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260 SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS	One LCS per batch of 20 or fewer samples	70-130% Recovery  <i>For projects requiring DoD QSM 4.1, DoD limits shall be used, unless otherwise specified by the project QAPP.</i>	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
MS/MSD	One MS/MSD per every 20 samples  <i>For projects requiring DoD QSM 4.1, one MS/MSD pair will be analyzed per preparatory batch per matrix if supplied with sufficient sample.</i>	Nominal limits of 70-130% are used as default limits. See also section 8.2 of this SOP.  <i>For projects requiring DoD QSM 4.1, DoD limits shall be used, unless otherwise specified by the project QAPP.</i>	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstration of Proficiency	Once per analyst initially; 4 reps of LCS and annually thereafter	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 2  
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-220-07	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	



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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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TABLE 3  
CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Chloromethane	50	52
Vinyl Chloride	62	64
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61, 63
Chloroform	83	85
Carbon Tetrachloride	117	119
Benzene	78	77, 51
1,2-Dichloroethane	62	98
Trichloroethene	95	97
Bromodichloromethane	83	85, 127
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
4-Methyl-2-pentanone	43	58
Tetrachloroethene	164	129, 131
Dibromochloromethane	129	127
1,3-Dichloropropene	76	78
1,2-Dibromomethane	107	109, 188
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
1,2,3-Trichloropropane	75	77, 110
1,4-Dichlorobenzene	146	111, 148
1,2-Dibromo-3-chloropropane	75	155, 157
Hexachlorobutadiene	225	223, 227

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TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

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TABLE 4  
INTERNAL STANDARD

INTERNAL STANDARD	TARGET ANALYTE
Pentafluorobenzene	Chloromethane
	Vinyl Chloride
	Trichlorofluoromethane
	1,1-Dichloroethene
	Chloroform
INTERNAL STANDARD	TARGET ANALYTE
1,4-Difluorobenzene	Carbon Tetrachloride
	Benzene
	1,2-Dichloroethane
	Trichloroethene
	Bromodichloromethane
	cis-1,3-Dichloropropene
	trans-1,3-Dichloropropene
	4-Methyl-2-pentanone
	1,2-Dibromomethane
INTERNAL STANDARD	TARGET ANALYTE
Chlorobenzene-d5	Tetrachloroethene
	Dibromochloromethane
	1,3-Dichloropropene
	1,1,1,2-Tetrachloroethane
INTERNAL STANDARD	TARGET ANALYTE
1,4-Dichlorobenzene-d4	1,1,2,2-Tetrachloroethane
	1,2,3-Trichloropropane
	1,4-Dichlorobenzene
	1,2-Dibromo-3-chloropropane
	Hexachlorobutadiene

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

TABLE 5  
STANDARD INFORMATION

VOA Standards

Standard	Concentration	Manufacturer	Catalog Number
1,2,3 Trimethylbenzene	2000 ug/mL	Restek	58733
1,2,3 Trichlorobenzene	2000 ug/mL	Accustandard	M-502-47-10X
1,2,4 Trimethylbenzene	2000 ug/mL	Accustandard	M-502-54-10X
1,3,5 Trichlorobenzene	neat	Supelco	44-2235
1,3,5 Trimethylbenzene	2000 ug/mL	Accustandard	M502-55-10X
2-CEVE	2000 ug/mL	Accustandard	M-601C-10X
502.2 Cal Mix #1 (gases)	2000 ug/mL	Restek	30042
502.2 Cal2000 Mega Mix	2000 ug/mL	Restek	30431
504.1 Cal Mix	200 ug/mL	Accustandard	M-504.1-CSS
Acrolein & Acrylonitrile	5000 ug/mL	Accustandard	M-603-M-5X
Appendix IX Volatiles Mix	various	Accustandard	M-8240C-R3-10X
Bromochloromethane	2000 ug/mL	Accustandard	M-502-03-10X
California Oxygenates Mix #1	2000 - 10,000 ug/mL	Restek	30465
Carbon Disulfide	2000 ug/mL	Restek	30258
Chloroprene	2000 ug/mL	Accustandard	APPX9-048-R1
Custom GC Std	2000 ug/mL	Accustandard	S-11160
Custom VOC mix	various	Accustandard	S-7920-R1
Custom Volatile GC/MS Std	2000 ug/mL	Accustandard	S-3432B
Custom Volatiles GC/MS	2000 ug/mL	Accustandard	S-3432A
Diethyl Ether	5000 ug/mL	Accustandard	AS E0285
Freon 113	2000 ug/mL	Supelco	4-7944
Method 8260 Additions	2000 ug/mL	Accustandard	M-8260-ADD-10X
Method 8260B-Revision	2000 ug/mL	Accustandard	M-8240B-R-10X
MTBE	2000 ug/mL	Supelco	4-8483
Napthalene	2000 ug/mL	Accustandard	M-502-40-10X
THF	2000 ug/mL	Accustandard	S-4575-10X
Vinyl Acetate	2000 ug/mL	Restek	30216
Vinyl Acetate	2000 ug/mL	Accustandard	APPX9-211-20X
VOA Calibration Mix #1 (Ketones)	5000 ug/mL	Restek	30006
TCL Ketone Mix	5000 ug/mL	Accustandard	CLP-022-25X
VOC Liquid Mix	2000 ug/mL	Accustandard	M-502A-R2-10X
Volatile Organic Compounds (gases)	2000 ug/mL	Accustandard	M-502B-10X
<b>IS/SS/Tune</b>			
Custom 8260 IS	5000 ug/mL	Restek	54577
Custom 8260 SS	5000 ug/mL	Restek	54578
4-BFB	2000 ug/mL	Supelco	48083
VOA Tuning Compound (BFB)	5000 ug/mL	Restek	30003
1,2 Dichlorobenzene-D4	2000 ug/mL	Supelco	48952-U
Fluorobenzene	2000 ug/mL	Supelco	
VOA IS (CLP)	2500 ug/mL	Restek	30004
VOA SS (CLP)	2000 ug/mL	Supelco	48943
624 IS	1500 ug/mL	Restek	30023
4-BFB/Fluorobenzene/Pentafl. (EPA 624)	20000 ug/mL	Accustandard	M-624-SS-M
8260A SS	2500 ug/mL	Restek	30240
<b>CLP Only</b>			
04.1 CLP VOA Cal 2000	2000 ug/mL	Restek	30456
LCS-IS	2500 ug/mL	Accustandard	CLP-LCS-IS-100X
LCS-Volatiles	200 ug/mL	Accustandard	CLP-LCS-V
CLP Volatiles DMC Stock Solution	deuterated compds	Cambridge Isotope	ES 5038
3.2 OLC mix	1000 - 2000 ug/mL	Restek	30492

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 1  
EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES  
GCMS-T INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 8/6/09 08:52  
Reviewed by/Date:

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD			Y/N	MS/MSD	PH	ANALYST	COMMENTS
					5030	5035	1311					
SO N/A BFB	IB406	-	-	PF8238A0				Y			HCG	
WST050706A	TSS33	1	1	T826A31				Y				
LCSA W6167M4-1	TSS34	1	2					Y				
VAKA	TSS35	1	3					N				
↓ B W6167M4-2	TSS36	1	4					Y				target with
SL4258-1	A TSS37	1	5		X			Y	1	42		
-6	B TSS38	1	6					Y	2	42		
SL4258-1	A TSS39	1	7					Y	3	42		
-6	A TSS40	1	8					Y	4	42		
SL4337-7	A TSS41	1	9					Y	5	42		
SL4258-2	B TSS42	1	10					Y	6	42		
-4	B TSS43	1	11					Y	7	42		
SL4351-2	B TSS44	1	12					N	8	42		1561 RR
-4	B TSS45	1	13					Y	9	42		
-7	B TSS46	1	14					Y	10	42		
SL4337-1	A TSS47	1	15					Y	11	42		
-2	A TSS48	1	16					Y	12	42		
-3	A TSS49	1	17					Y	13	42		
-4	C TSS50	1	18					Y	14	42		
9-6	C TSS51	1	19					Y	15	42		
9-6-5	E TSS52	1	20					Y	16	42		
SL4422-1	A TSS53	1	21					Y	17	42		
-2	A TSS54	1	22					Y	18	42		20:2A ✓
RINSE	TSS55	Z	23					-	-	-		
↓	TSS56		24					-	-	-		

QAMS413

STANDARD	CODE	STANDARD	CODE
BFB	V2802	IS MIX	V2810
CAL STD.	V2808	SS MIX	V2811
LCS/MS MIX	V2809		
EXTRAS MIX	V2797		

0000017

Circle Methods:  
SW846 8260  
EPA 624  
EPA 524  
SIM

OLM 04.2  
OLM 03.1  
OLC 02.1  
OLC 03.2




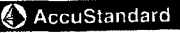

(44) 8/7/09

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 2  
EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

**KATAHDIN ANALYTICAL SERVICES**  
STOCK STANDARDS RECEIVED

GC/MS LABORATORY  
REVIEWED BY/DATE:

AMP 1514 1519 1520 1521	1-Chlorohexane (EPA-1208) Lot: CB-8341A Ultra Exp: 11/30/09 1200 µg/mL	Rec'd 11/07/07
AMP 1522 1523 1524 1525	 Cat# 90494 MS-1000 Single in Purge and Trap Method Lot # AD40187 Exp: 11/12 Store: Freezer Prestek Corporation 110 Banner Circle - Bensalem, PA 19023	Rec'd 11/7/07 FRL
AMP 1526 1527	 Cat# 90465 Diffusion Degradable Glass P 230-1000 Single in Purge and Trap Method Lot # AD49252 Exp: 12/11 Store: Freezer Prestek Corporation 110 Banner Circle - Bensalem, PA 19023	Rec'd DMF 11/20/07
AMP 1528 1529	 AS-E0285 Diethyl ether 5000 µg/mL in MeOH Lot: B4070099-1A Exp: Jan 19, 2010	Rec'd DMF 11/21/07
AMP 1530 1531	 CLP-LC-IS-100X Laboratory Control Sample - Internal Standard Mix 2500 µg/mL in MeOH Lot: B2090027 Exp: Sep 8, 2012	Rec'd DMF 11/26/07
AMP 1532 1533	 Cat# 90624 ON 101 MS-1000 Single in Purge and Trap Method Lot # AD50631 Exp: 3/10 Store: Freezer Prestek Corporation 110 Banner Circle - Bensalem, PA 19023	Rec'd DMF 11/26/07

TITLE: ANALYSIS OF VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP GC/MS:  
SW-846 METHOD 8260 – MODIFIED FOR SELECTED ION MONITORING (SIM)

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES  
GC/MS VOA STANDARD PREP LOG BOOK

CODE	STOCK NAME	STOCK	STOCK CONC UG/ML	VOLUME ADDED
V2809	50:2 Cal Mix #1	AMP 2187	2000	50
STANDARD: 8260 LCS	MTBE	AMP 2121		
FINAL CONC (ug/mL): 200	50:2 Cal Mix #1	AMP 2143		
FINAL VOLUME (mL): 1.5	5:4575-10x	AMP 2147		
PREP DATE: 8/14/09	M-8260-ADD-10x	AMP 2180		
EXPIRATION DATE: 8/18/09	1-Chloro-o-xylene	AMP 2109	1000	50
MEOH VOLUME (uL): 150	cyclohexane	AMP 2183		
MEOH LOT # E25806				
INITIALS: HCN				
8/14/09 (HCN)				
V2810	Custom 8260 IS Mix	AMP 2071	5000	50
STANDARD: 8260 IS "I"				
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/15/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4950				
MEOH LOT # E25806				
INITIALS: HCN				
8/15/09 (HCN)				
V2811	8260 Sample Mix	AMP 2223	2500	100
STANDARD: 8260 56 "I"				
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/15/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4900				
MEOH LOT # E25806				
INITIALS: HCN				
8/15/09 (HCN)				

Reviewed by/Date:

**KATAHDIN ANALYTICAL SERVICES, INC.**  
**STANDARD OPERATING PROCEDURE**

**SOP Number: SD-902**  
**Revision History**  
**Cover Page**  
**Page 1**

**TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002  
 Approved By: \_\_\_\_\_  
 Group Supervisor: Andrea Colby Date: 6/6/02  
 Lab Operations Mgr: J. C. Burton Date: 6/5/02  
 QA Officer: Deborah J. Nadeau Date: 6/6/02

**Revision History:**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.15 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6/6/02	6/6/02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. updated new incoming	DN	05/04	05/04
06	Added procedure + Logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA soil freezer storage.	DN	01-26-04	01-26-04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures Band 10. Updated Table and Figures w/ current ones. Added wording to Sect. 7.7.5 to clarify how pH measurements are taken.	LAID	02/07	02/07
08	Added Summary Stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRQR. Other minor changes.	DN	05/09 08/09 8.4.09	05/09 08/09

Added section concerning locking of coolers. Added more detail to 7.18 on unique container IDs. Added more detail on immediate COCs & a section on retention of samples.

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

---

I acknowledge receipt of copy \_\_\_\_ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_



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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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## **1.0   SCOPE AND APPLICATION**

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

### **1.1   Definitions**

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

### **1.2   Responsibilities**

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability"), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify

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TITLE:       SAMPLE RECEIPT AND INTERNAL CONTROL

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problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

### 1.3    Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

### 1.4    Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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Standards," current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

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## **2.0   SUMMARY OF METHOD**

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

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## **3.0   INTERFERENCES**

Not applicable.

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## **4.0   APPARATUS AND MATERIALS**

- 4.1   Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).
  - 4.2   Capillary tubes – 75 mm Hematocrit Tubes, disposable
  - 4.3   Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.
  - 4.4   Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.
  - 4.5   Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.
- 

## **5.0   REAGENTS**

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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## **6.0     SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

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## **7.0     PROCEDURES**

### **PROCEDURES FOR SAMPLE CUSTODIAN**

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

- 7.1     When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).
- 7.2     Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).
- 7.3     Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 4). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.
- 7.4     Inventory the COCs for any "RUSH" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO <sub>2</sub>
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5   Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 6). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6   If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 7 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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- 7.7     Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

- 7.8     Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.
- 7.9     Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

- 7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.
- 7.10.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.
- 7.10.2 A vacuum pump is attached.
- 7.10.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.
- 7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.
- 7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.
- 7.11 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.
- 7.12 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 9).
- 7.13 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.
- 7.14 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15   Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.16   When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17   The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -           Enter client sample description.

ReceiveDate -       Enter in date that samples were received in the lab in the format YY-Month-DD.

CollectDate -       Enter in date that samples were collected in the format YY-Month-DDTIME.

TAT -                Enter TAT for hardcopy report.

DueDate -           Due date will automatically be calculated based on calendar days.

VerbalDate -        Manually type in verbal due date.



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TITLE:       SAMPLE RECEIPT AND INTERNAL CONTROL

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QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.
Account Name -	Account name will automatically be entered.
Collected By -	Enter name/initials of sampler listed on COC. If unknown, enter "Client".
Locator -	May be used for client ID information when requested by the project manager.
Site -	Enter project site name.
Description -	May be used for long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
Matrix -	Enter sample matrix code where  AQ = Aqueous                      SLD = Food Solid SL = Solid, Soil, Sludge        AR = Air FP = Free Product               SWAB = Swab WP = Wipe                        SAL = Saline NOAQ = NonAqueous              TIS = Tissue DW = Drinking Water
Product Code -	Enter analysis code per test requested on COC.
Type -	Product code type will automatically be entered where S = Stand alone

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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P = Parent  
C = Children

Fact. -	No entry-default is 1.
Price -	This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.
Cost -	No entry needed.
Lev -	No entry needed.
Type -	Container type will automatically be entered.
Bot -	Enter number of containers per test for printing of labels.
Login Info -	Parameter Data Screen will open. Enter following information
	KAS Proj. Manager- Initials of Katahdin person overseeing the project.
	Client PO#- Client purchase order.
	Project- Project name.
	Cooler Temperature- Temperature blanks or cooler temps.
	Delivery Services- Method of delivery to the lab.
	QC Level- QC Level of report and regulatory agency (ie., IV NFESC).
	SDG ID- Sample Delivery Group ID if applicable.
	SDG Status- Begin, Continue or End.
	Analysis Instructions- PM will enter special instructions regarding project.
	Report Instructions- PM will enter special instructions regarding project.
	Regulatory List- Used for federal programs.
	EDD Format- Specific KAS EDD format.
	Select "SAVE" and then "CANCEL".
Addresses -	Select "Addresses" and the Address Links screen will open. The billing address is the default address of the

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TITLE:       SAMPLE RECEIPT AND INTERNAL CONTROL

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account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.17.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.18 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDXL)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".

Note: As stated in "create containers" above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-001(a), SC9001-001(b), SC9001-001(c)

Example: One job containing two client samples with 2 different containers for each:

SC9002-001(a), SC9002-001(b), SC9002-002(a), SC9002-002(b)

7.19 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

- 7.20.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.
- 7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.
- 7.20.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in "VOA Refrigerator 2" located in the Volatiles Laboratory.
- 7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.
- 7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a "paint can" and stored in the walk-in.
- 7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in "VOA Freezer 1" in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented with respect to native samples, extracts and digestates within the laboratory. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

- 7.21 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and

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TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

- 7.22 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 13).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

#### PROCEDURES FOR CHEMISTS

- 7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.
- 7.24 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 14) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. When the logbook for Immediate Internal COC's is used, the standard internal COC's do not have to be signed at a later date. The Immediate Internal COC Logbook must always be consulted if there is ever a question about whether an internal COC has been completed.

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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- 7.25   If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.
- 7.26   If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.
- 7.27   After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

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## 8.0    QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

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## 9.0    METHOD PERFORMANCE

Not applicable.

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## 10.0   APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Inc., Environmental Health & Safety Manual, current revision.

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## LIST OF TABLES & FIGURES

Table 1	Sampling and Preservation Requirements
Figure 1	Example of Standard Katahdin Chain-of-Custody Form

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TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

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LIST OF TABLES & FIGURES, cont'd

Figure 2	Example of Homeowner Katahdin Chain-of-Custody Form
Figure 3	Example of Food/Microbiology Katahdin Chain-of-Custody Form
Figure 4	Example of Sample Receiving Logbook
Figure 5	Example of Wet Chemistry Shorts and Rushes Logbook
Figure 6	Example of Katahdin Sample Receipt Condition Report
Figure 7	IR Thermometer Manufacturer's Instructions for Changing Emissivity
Figure 8	Example of Sample Filtration Logbook
Figure 9	Measured Turbidity and Preservation of Incoming Samples Logbook
Figure 10	Example of Laboratory Incoming Sample Information Sheet (ISIS)
Figure 11	Example Katahdin Work order/login COC Report
Figure 12	Example of Katahdin Internal Chain-of-Custody Form
Figure 13	Example of Refrigerator Temperature Logbook
Figure 14	Example of Immediate Internal COC Logbook

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1  
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GENERAL CHEMICAL ANALYSES</b>					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO <sub>4</sub> , NO <sub>2</sub> , NO <sub>3</sub> )	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO <sub>2</sub>	SM4500-CO <sub>2</sub> C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2, SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate, Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS), Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GENERAL CHEMICAL ANALYSES</b>					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
<b>ELEMENTAL ANALYSES</b>					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
<b>GC ORGANIC ANALYSES</b>					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GC ORGANIC ANALYSES</b>					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
<b>GC/MS ORGANIC ANALYSES</b>					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
<b>HPLC ANALYSES</b>					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
<b>MICROBIOLOGICAL ANALYSES</b>					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GENERAL CHEMICAL ANALYSES</b>		<b>4 oz=100 g</b>			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest., Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate, Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest, Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
<b>ELEMENTAL ANALYSES</b>					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS Elements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)  
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>ELEMENTAL ANALYSES (cont.)</b>		<b>4 oz=100 g</b>			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30days/24hrs
<b>GC ORGANIC ANALYSES</b>					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
<b>HPLC ANALYSES</b>					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
<b>GC/MS ANALYSES</b>					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatle Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatle Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>RCRA - HAZARDOUS WASTE CHARAC.</b>					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
<b>TCLP</b>					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 mL 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

SOP Number: SD-902-08  
Date Issued: 08/09  
Page 24 of 37

FIGURE 1

[illegible]


THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS.

## ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

## EXAMPLE OF HOMEOWNER KATAHDIN CHAIN-OF-CUSTODY FORM



**Katahdin**  
ANALYTICAL SERVICES

600 Technology Way  
P.O. Box 540  
Scarborough, ME 04070  
Tel: (207) 874-2400 Fax: (207) 775-4029

# Homeowner Chain of Custody

Client:		Contact:		Phone:		Fax:	
Address:		City:		State:		Zip:	
Purchase Order #:		Project Name/No.:		E-mail:			
Billing Address (if different):							
Sampler (Print/Sign):				Copies To:			
*** Test results are for compliance and will be reported to the state (see statement below).      yes      no      Compliance samples must be received on ice.							
Lab Use Only		Work Order #:		KAS Project Manager:		Requested Services	
Shipping:		UPS	Fed-Ex	Mail	Drop-Off		
Sample(s) Received on Ice?		Yes	No	Temperature if Iced:			
Sample Description (Sample Identification and/or Lot #)		Date Collected	Time Collected	No. of Cntrs.	Standard	Asenic	Total Coliforme
					Lead	Safety Test - Coliform & NPH	FHA/MSH
						Fluoride	Uranium
							What's Included in the Standard Test and the FHA/MSH Test.
							Standard Homeowner
							Total Coliform/e-coli
							Nitrate, Nitrite
							Chloride, pH
							Hardness
							Copper, Iron
							Manganese
							Sodium
							FHA/MSH
							Standard plus
							Lead
							Turbidity
							Color
							Odor
Relinquished By:		Date/Time:	Received By:		Relinquished By:		Date/Time:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at <6 degrees celcius. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements.

SOP Number: SD-902-08  
Date Issued: 08/09  
Page 26 of 37

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

### EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM

[illegible]



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By: \_\_\_\_\_

Date: \_\_\_\_\_

Reviewed By: \_\_\_\_\_

Date: \_\_\_\_\_

0000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES					Receipt Date:												Comments (Quick TAT, MS/MSD, etc.)
HOLDING TIME				Rush Parameters	Immediate			24 Hr		48 Hr							
Work Order Client	Matrix	Earliest Sampling Date	Earliest Sampling Time		pH	DO	Sulfide	Fe +2	Cu+6	Total BOD	Carbon BOD	Color	Nitrate	Nitrite	OP04	Set Solids	
_____																	
_____																	
_____																	
_____																	
_____																	
_____																	
_____																	
_____																	
_____																	
_____																	

0000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc.				Sample Receipt Condition Report	
Client:		KAS PM:		Sampled By:	
Project:		KIMS Entry By:		Delivered By:	
KAS Work Order#:		KIMS Review By:		Received By:	
SDG #:	Cooler: _____ of _____	Date/Time Rec.:			

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If not, has the cooling process begun (i.e. ice or packs present) and sample collection times <2hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: <b>Aqueous:</b> No bubble larger than a pea <b>Soil/Sediment:</b> Received in airtight container? Received in methanol? Methanol covering soil?					
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2 Sulfide - >9 Cyanide – pH >12					

\* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

**MODE Button Functions**

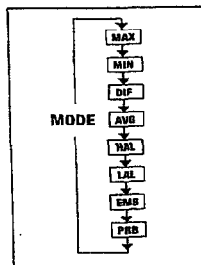
Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)\*, and Average (AVG)\*\* temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected.

Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.

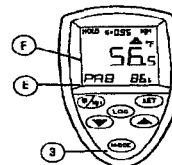
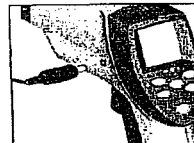
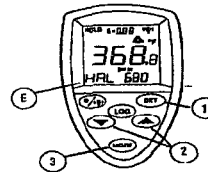
**Note:** PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

**Selecting a Function**

To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.



English



**Setting the High Alarm, Low Alarm, and Emissivity**

To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

**Using a Probe (PRB)**

Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

**Note:** PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

**TITLE:       SAMPLE RECEIPT AND INTERNAL CONTROL**

FIGURE 8

## EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

**KATAHDIN ANALYTICAL SERVICES, INC.**  
**Sample Filtration Logbook**

[illegible]

Reviewed and Approved by: \_\_\_\_\_ Date: \_\_\_\_\_

**QAAA097**

0000001

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

[illegible]

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)


KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:		ATTENTION:	
ADDRESS:			
TELEPHONE:		FAX:	
OTHER CLIENT CONTACTS:			
REPORT CC:		PM/SALES CONTACT:	
PO #:		CLIENT ACCOUNT:	
KAS QUOTE #:		KIMS QUOTE #:	
PROJECT NAME:		KIMS PROJECT REF.:	
SDG:		APPLICABLE CERTIFICATION:	
RECEIPT DATE:		MEANS OF DELIVERY:	
METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)
SUBCONTRACT PARAMETER		SUBCONTRACT LABORATORY	
QC LEVEL:		EDD:	
RUSH/VERBAL TAT:		HARDCOPY TAT:	
HISTORY:			
OTHER:			

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

 **Katahdin Analytical Services**  
Login Chain of Custody Report (Ino1)  
Jan. 26, 2007  
03:51 PM

Page: 1 of 1

**Login Number: SA0395**  
Account: KATAHD001  
Katahdin Analytical Services

**Web**

**Project:**

**Primary Report Address:**  
Leslie Dimond  
Katahdin Analytical Services  
600 Technology Way  
P.O. Box 540  
Scarborough, ME 04070

**Primary Invoice Address:**  
Accounts Payable  
Katahdin Analytical Services  
600 Technology Way  
P.O. Box 540  
Scarborough, ME 04070

**Report CC Addresses:**

**Invoice CC Addresses:**

**Login Information**

ANALYSIS INSTRUCTIONS :  
CHECK NO. :  
CLIENT PO# :  
COOLER TEMPERATURE : n/a  
DELIVERY SERVICES : In-House  
EDD FORMAT :  
MAIL DATE :  
PM : LAD  
PROJECT NAME : QC Holding Blanks  
QC LEVEL : I  
REGULATORY LIST :  
REPORT INSTRUCTIONS :  
SDG ID :  
SDG STATUS :

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	Verbal PR Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07		08-FEB-07	
Matrix: Aqueous	Product: 5 SW/260FULLSML	Hold Date (skipped): 09-FEB-07	Bottle Type:	Bottle Count: 2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07		08-FEB-07	
Matrix: Aqueous	Product: 5 SW/260FULLSML	Hold Date (skipped): 08-FEB-07	Bottle Type:	Bottle Count: 2		

**Total Samples: 2**      **Total Analyses: 2**



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

### EXTRACTIONS (AQ)

Work Order #: \_\_\_\_\_

[illegible]

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

### EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

### SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

**Corrective Action:** Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

[illegible]

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.  
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930	→ 0935	DSJ	yes <u>no</u>
Jacobs		WW4883-1A	ICP	9/14/06 0100	→ 1000	DSJ	yes <u>no</u>
CES		WW4965	BOD	9/20/06 0900	9/20/06 1000	CP	yes no
CCAB		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GENF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900	→ 1000	DSJ	yes <u>no</u>
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1005	CP	yes <u>no</u>
Highmer		WW4992	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PSEUDONITR PH, SP, ORP	9/21/06 1100	9/21/06 1257	DSJ	yes <u>no</u>
WTC		WW5001	BOD	9/21/06 1300		CP	yes no
Arizans		WW5016	NIT	9/22/06 1300	9/22/06 1100	DSJ	yes <u>no</u>
RANDSON		WW5010	↓	↓	↓	↓	yes <u>no</u>
EconHaine		WW5029	BOD	9/22/06 1100		CP	yes no

0A0C143

0000085

**KATAHDIN ANALYTICAL SERVICES, INC.**  
**STANDARD OPERATING PROCEDURE**

**SOP Number: SD-903**  
**Revision History**  
**Cover Page**  
**Page 1**

**TITLE: SAMPLE DISPOSAL**

Prepared By:

Whitlow

Date:

2/01

Approved By:

Group Supervisor:

Date:

Operations Manager:

John C. Banta

Date:

2/01

QA Officer:

Deborah J. Nadeau

Date:

2.01

General Manager:

Dennis E. Keefe

Date:

2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, added updated log-book and greater detail on disposal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regulations + to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

SOP Number: SD-903-04  
Date Issued: 05/09  
Page 2 of 17

---

TITLE:       SAMPLE DISPOSAL

---

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

---

I acknowledge receipt of copy \_\_\_\_ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

TITLE:       SAMPLE DISPOSAL

---

## 1.0   SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

### 1.1   Definitions

Hazardous Waste – A "Solid Waste" which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

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TITLE:       SAMPLE DISPOSAL

---

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

## 1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

---

TITLE:           SAMPLE DISPOSAL

---

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

---

## **2.0   SUMMARY OF METHOD**

Not applicable.

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## **3.0   INTERFERENCES**

Not applicable.

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## **4.0   APPARATUS AND MATERIALS**

Not applicable.

---



---

TITLE:       SAMPLE DISPOSAL

---

## **5.0    REAGENTS**

Not applicable.

---

## **6.0    SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Not applicable.

---

## **7.0    PROCEDURES**

- 7.1    Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.
- 7.2    Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.
- 7.3    The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3<sup>rd</sup> party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.
- 7.4    Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3<sup>rd</sup> party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

---

TITLE:       SAMPLE DISPOSAL

---

- 7.5   Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, choose a section that needs extra space the most and remove the oldest samples.

***Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handling samples during disposal***

- 7.6   Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles(VOA)
VOA (SL)	Solid Volatiles(VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

- 7.7   The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

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TITLE:           SAMPLE DISPOSAL

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If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8     As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9     Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1   Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2   Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream "K". Containers are disposed of with general trash. Solid samples are placed into waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.3   Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream "A". Containers are disposed of with general trash. Solid samples are placed in waste stream "L" with their containers. The disposal date is recorded in the SDL.

7.9.4   Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)

Refer to section 7.10 below.

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TITLE:       SAMPLE DISPOSAL

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7.9.5   Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6   Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "Q". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "F" with their containers. The disposal date is recorded in the SDL.

7.9.7   Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "O". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.8   Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "NHi". Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9   Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream "A". There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the "O" waste stream. There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. Lastly, samples marked "free product" on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10   Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

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TITLE:           SAMPLE DISPOSAL

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- 7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.
- 7.10.2 This process is continued until the pH is between 7 and 8. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.
- 7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.
- 7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.
- 7.11 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a "lab pack" of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.
- 7.12 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection  
Bureau of Remediation & Waste Management  
State House Station #17  
Augusta, ME. 04333  
Attn: Annual Hazardous Waste Report

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TITLE:           SAMPLE DISPOSAL

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## **8.0     QUALITY CONTROL AND ACCEPTANCE CRITERIA**

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

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## **9.0     METHOD PERFORMANCE**

Not applicable.

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## **10.0    APPLICABLE DOCUMENTS/REFERENCES**

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

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## **LIST OF TABLES AND FIGURES**

- |          |  |
|----------|--|
| Figure 1 | Example of Sample Disposal Logbook                           |
| Figure 2 | Example of KIMS Generated Waste Disposal Report              |
| Figure 3 | Example Of Hazardous Waste Area Daily Check Documentation    |
| Figure 4 | Characteristic Toxic Hazardous Waste and TCLP concentrations |
| Figure 5 | Example of Elementary Neutralization Logbook                 |

TITLE: SAMPLE DISPOSAL

FIGURE 1  
EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. -SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	CRITERIA	SAMPLE KNOWLEDGE										DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS					
SA5783-1	WC	10-17-07	✓											1-22-08	GN
SA5786-1		10-17-07	✓												
SA5787-1,2,4		10-17-07	✓												
SA5790-1		10-19-07		✓											
SA5793-1		10-17-07	✓												
SA5795-1-9		10-23-07	✓												
SA5797-1		10-23-07	✓												
SA5798-1,2		10-23-07			✓										
SA5799-1-5		10-23-07	✓												
SA5804-1,2		10-23-07	✓												
SA5807-1,2		10-23-07	✓			2				2					
SA5810-4		10-23-07	✓												

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TITLE: SAMPLE DISPOSAL

FIGURE 2  
EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000  
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value
SA6605-1		NEED	12/02/07		
SA6606-1		NEED	12/02/07		
SA6607-1		NEED	11/15/07		
SA6608-1		NEED	12/06/07	ORG	1.17 MG/L (HIGH)
SA6608-1		NEED	12/06/07		
SA6608-2		NEED	12/06/07	AA	13 MG/KG (HIGH)
SA6609-1		NEED	11/26/07		
SA6609-1		NEED	11/26/07		
SA6610-1		NEED	11/30/07		
SA6611-1	FCS-020	NEED	12/07/07		
SA6611-2	FCS-020	NEED	12/07/07		
SA6611-3	FCS-020	NEED	12/07/07		
SA6611-4	FCS-020	NEED	12/07/07		
SA6611-5	FCS-020	NEED	12/07/07		
SA6611-6	FCS-020	NEED	12/07/07		
SA6611-7	FCS-020	NEED	12/07/07		
SA6611-8	FCS-020	NEED	12/07/07		
SA6612-1	NSA-030	NEED	12/07/07		
SA6612-2	NSA-030	NEED	12/07/07		
SA6612-3	NSA-030	NEED	12/07/07		
SA6612-4	NSA-030	NEED	12/07/07	ORG	1.70735 MG/L (HIGH)
SA6612-5	NSA-030	NEED	12/07/07	ORG	1.0481 MG/L (HIGH)



TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for  
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 2008

QA00315

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" state?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No initials)	John H. H.	John H. H.	John H. H.	John H. H.	John H. H.
9. Time of inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of inspection (Name/Date)	John H. H. 1-28-08	John H. H. 1-28-08	John H. H. 1-28-08	John H. H. 1-28-08	John H. H. 1-28-08
Deficiency notes:					
Corrective action:					
By (Name/Date):					

0000060

TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

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TITLE:       SAMPLE DISPOSAL

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FIGURE 4, cont'd

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 3-4-09		Time: 12.00	Analyst: GA
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

25  
240

Date: 3-10-09		Time: 13:45	Analyst: GA
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

49  
281

## APPENDIX 2

## **APPENDIX 2**

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### **Project Sampling SOPs**

## **STANDARD OPERATING PROCEDURE FOR WELL INTEGRITY TESTING**

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### **PURPOSE AND SCOPE**

This standard operating procedure (SOP) describes the method for performing well integrity testing. Prior to collecting samples or measuring water levels in existing wells which have not been included in the long-term monitoring for the Northeast Quadrant groundwater remedy, well integrity tests will be conducted. These tests will include visual inspection of the well, depth measurement to confirm well depth and a slug test to confirm the hydraulic connection of the well to the saturated unconsolidated deposits. In addition, the position of the water table in relation to the well screen will be noted.

### **WELL INTEGRITY TESTING**

Integrity testing will be conducted in the following manner. The historical well construction data will be used to make a comparison in the field of well diameter and depth of installation. The depth will be determined by sounding the well to the bottom with a weighted steel or fiberglass tape. The water level will be measured with an electronic water level sensor. A known volume slug will then be added to the well and the water level change immediately recorded. The water level will be monitored for up to 48 hours or until 50 percent of the induced water level change has recovered, whichever is less. All tapes, slugs and water level sensors used for the well integrity tests shall be decontaminated between wells. Decontamination procedures for sampling equipment should be followed (Groundwater Sampling SOP).

Should a well not meet the hydraulic recovery of 50 percent recovery in 48 hours, an attempt to develop the well will be made and the hydraulic recovery test redone. Development should be done using a bailer to surge and purge the well. If possible, continue development until clear, sediment-free water is obtained. All PPE, development water, decontamination fluids and other residuals generated during the well integrity testing will be managed in accordance with the Materials Management SOP. The results of the well integrity testing will be recorded on the well integrity test form provided.

## Well Integrity Test

Well Name: \_\_\_\_\_

Well Location: \_\_\_\_\_

Date: \_\_\_\_\_

### Well Construction Specification

	Historic Record	Measured Today
Total Depth of Well from Surface (feet):	_____	_____
Stick-up (feet):	_____	_____
Well Casing Type:	_____	_____
Well Diameter:	_____	_____

### Checklist

New Lock Installed	<input type="checkbox"/>
New Label Added	<input type="checkbox"/>
Slug/Bailer/Water Level Tape	<input type="checkbox"/>
Decontaminated	<input type="checkbox"/>
Flagging Tape Added	<input type="checkbox"/>
Well Development Necessary	<input type="checkbox"/>

### Measuring Point

Existing Mark	<input type="checkbox"/>	Created New Mark	<input type="checkbox"/>
Measuring Point Location:	_____		

Condition of Protective Casing and Seal: \_\_\_\_\_

Position of Water Table Relative to Well Screen: \_\_\_\_\_

### Water Level Response

Beginning Water Level (feet):	_____
Slug <input type="checkbox"/>	Bailer <input type="checkbox"/>

Displacement Volume (gallons): \_\_\_\_\_

Time	Water Level	Comments
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

If well development was necessary, describe method: \_\_\_\_\_

Name: \_\_\_\_\_ Company: \_\_\_\_\_



## **STANDARD OPERATING PROCEDURE FOR MONITORING WELL INSTALLATION**

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### **PURPOSE AND SCOPE**

This standard operating procedure (SOP) describes the procedures to install groundwater monitoring wells using the rotosonic drilling method. The wells shall be drilled to a depth of approximately ten feet below the water table. It is anticipated that monitoring well depths will be 15 to 30 feet below ground surface. Some monitoring wells may extend below the bedrock surface. Monitoring wells are to be installed such that the water table is within the screened interval of the well. During monitoring well drilling, collect continuous samples of subsurface material to the required depth of the boring, or as directed by the supervising geologist using the rotosonic core barrel. Collect a sub-sample from each five-foot interval within the unconsolidated deposits and place the sample into a jar for headspace screening with a Photo-Ionization Detector (PID). Describe the unconsolidated deposits sample using the Unified Soil Classification System. For rock core samples, describe the rock type, fracturing and any weathering. The rotosonic casing shall be advanced after an interval has been sampled.

If boulders or obstructions are encountered during drilling, advance the casing past or through the obstacles by either drilling or coring. If the borehole cannot reasonably be advanced or the obstruction encountered is near the surface, start another hole no more than 20 feet from the failed hole.

Personnel will also be responsible for documenting drilling events in the field boring log or and notebook. The drilling contractor is responsible for obtaining accurate and representative samples, informing the supervising geologist of changes in drilling pressure and loss of circulation, and keeping a separate general log of soils or bedrock encountered.

### **UNCONSOLIDATED DEPOSIT AND BEDROCK SAMPLING PROCEDURES**

#### ***Unconsolidated deposits***

Step 1: Obtain gross sample with the rotosonic core barrel.

Step 2: Personnel shall record the following information on the field boring log or in the field note book:

- Soil description by the Unified Soil Classification System. Each stratum shall be identified in the following order:
  - Color
  - Soil type
  - Classification symbol
  - Consistency or relative density
  - Moisture
  - Structure (if any), and

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**SOP 2**  
**Revision Date:** 3/25/10  
**Revision Number:** 0

- Modifying information such as grain size and percentages, particle shape, cementation, plasticity, and stratification
  - Recovery
  - Moisture content
  - Other observations
- Step 3 Headspace screening (see below).

#### ***Rock Core***

Step 1 Obtain gross rock core sample with the roto sonic core barrel.

Step 2 Personnel shall record the following information on the field boring log or in the field note book:

- Description of rock type
- Description of fractures
- Description of weathering
- Other observations

### **Unconsolidated Deposit and Bedrock Logging**

All drilling and well installation operations must be observed by a geologist or engineer. The encountered subsurface conditions shall be recorded on a Field Boring Log and by documenting other necessary information in a Field Notebook

The Field Boring Log shall be filled out completely in legible printing, using ink. These logs are original documents, and shall not be erased. If corrections are needed, the information shall be crossed out with a single line, initialed and dated, and the amended information shall be noted. The following data shall be recorded on the Field Boring Log, at a minimum:

**Boring Location:** A sketch of the boring location shall be presented on the first sheet of each log that includes sufficient detail to allow the boring to be found.

**Depth:** Depths recorded on the log should be measured from ground surface. If an alternate reference point is used, the elevation, description, and distance above or below ground surface shall be noted.

**Geologic classification:** The geologic classification shall be noted at each unconsolidated deposit or rock type change. Additional information regarding the presence of gravel, roots, wood, organic material, etc., shall be entered at the appropriate depth.

**Drilling Details:** Soil moisture changes, caving soil, odors, color change, changes in drill rig response and other salient details shall be noted. Comments made by the drill rig operator regarding drilling conditions shall be noted.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**SOP 2**  
**Revision Date:** 3/25/10  
**Revision Number:** 0

### **Head Space Screening**

Perform field screening for volatile organic compounds (VOCs) using a portable Photo-Ionization Detector (PID) or Flame-Ionization Detector (FID) using the following procedure:

1. PID and FID field instruments shall be operated and calibrated to yield "total organic vapors" in ppm. PID instruments must be operated with a 10.6 or 11.7 eV (+/-) lamp source. Operation, maintenance, and calibration shall be performed in accordance with the manufacturer's specifications.
2. Half fill a clean glass jar with the sample to be screened. Quickly cover the open top with two sheets of clean aluminum foil and subsequently apply screw cap to tightly seal the jar.
3. Vigorously shake the jar for 15 seconds.
4. Allow headspace to develop for at least 10 minutes. (Where ambient temperatures are below 32° F headspace development should be within a heated vehicle or building.)
5. After 10-minute headspace development period, again vigorously shake the jar for 15 seconds.
6. Remove screw lid to expose foil seal. Quickly puncture the foil seal with the instrument sampling probe, to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particulates.
7. Following probe insertion through the foil seal, record the highest meter response for the jar headspace concentration. Maximum response should occur between two and five seconds. (Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case headspace data should be discounted.)

### **WELL INSTALLATION**

Following drilling at each location, a 2-inch diameter monitoring well shall be installed. The 2-inch diameter monitoring well shall be constructed using the following materials:

- 2-inch-diameter, flush-threaded, Schedule 40 polyvinyl chloride (PVC) blank casing;
- 2-inch-diameter, flush-threaded, Schedule 40 PVC, 0.010-inch slotted (10 slot) screen;
- 2-inch-diameter, flush-threaded, Schedule 40 PVC end cap;
- Pre-washed silica sand filter pack (appropriately sized); and
- Cement-bentonite grout.

Monitoring wells are to be installed such that the water table is within the screened interval of the well. If material is found to be in the casing, clean the borehole out to the bottom.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**SOP 2**  
**Revision Date:** 3/25/10  
**Revision Number:** 0

If the borehole has been advanced to a depth greater than the bottom of the well to be installed, backfill the borehole to the appropriate depth with sand. While placing the sand, measure the depth to the top of the sand often with a weighted tape measure until the sand has reached the desired depth. The well casing and screen shall be set in place by lowering it through the boring and placing it at the desired depth.

Filter pack shall be placed to the bottom of the annulus. Depth measurements to the bottom of the annular space shall take place as the sand is added. Sand shall be added until the sand pack is 1 foot above the top of the screen and the well screen shall be surged with a bottom-filling bailer to seat the sand pack. Additional sand pack shall be added to a maximum of 2 feet above the top of the well screen. Grout the remainder of the annular space using cement/bentonite mixture containing two to ten percent bentonite by weight. The grout shall be tremied to the desired depth.

The new groundwater monitoring wells shall have a flush-mount well traffic box completion. Elements of the flush-mount surface well completion shall include the following:

- A traffic rated flush-mount traffic box;
- A concrete collar and apron; and
- A locking j-plug.

## **DOCUMENTATION**

During the installation of each monitoring well, a series of measurements shall be taken and recorded. These measurements shall include:

- Depth of borehole;
- Length of tail pipe or sump (if used);
- Length of screen and type of slots (if used);
- Length of riser (if used);
- Total length of well to ground surface;
- Depth to stabilized water level; and
- Depth to top of bedrock, if the well is installed into bedrock.

Other data that shall be included are the screen and riser materials, diameters of components, type and thickness of sand packs, types and thicknesses of grout materials, riser stick-up above ground, description of the measuring point and description of the well location.

All data shall be recorded on the Field Boring Log. A field book can be used as additional means of recording data. All documentation shall remain in the project files.

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**SOP 2**  
**Revision Date:** 3/25/10  
**Revision Number:** 0

## **WELL DEVELOPMENT**

Upon completion of monitoring-well installation it is generally necessary to proceed with well development to ensure that there is hydraulic connection between the well screen and the formation. Direct pumping or pumping with the use of a surge block or other effective means shall be used for well development.

Well development by direct pumping is most effective in monitoring wells that will yield water continuously. Effective development cannot be accomplished if the pump has to be shut off to allow the well to recharge.

Set the intake of the pump (if a submersible is used) or the end of a drop line in the center of the saturated portion of the screened interval of the monitoring well. Pump from the well while raising and lowering the drop line or pump. This motion of the pump or intake line will act to a limited extent as a surge block to improve the removal of fines from around the screen. Continue pumping from the well until clear, sediment-free water is obtained.

Well development using a surge block shall proceed by lowering the surge block to the water level in the well. Start the surge action above the screen by slowly raising and lowering the surge block (three to five feet) allowing the water column to transmit the surge action to the screened interval. A slow initial surging will allow material which is blocking the screen to separate and become suspended.

After a number (five to ten) of plunger strokes, remove the surge block and purge the well using a pump. The returned water may contain suspended silt and clay particles. As development continues, slowly increase the depth of surging to the bottom of the well screen. For monitoring wells with long screens (greater than ten feet) surging should be undertaken along the entire screen length in short intervals (two to three feet) at a time. Continue this cycle of surging and purging until clear, sediment-free water is obtained.

## **DECONTAMINATION OF EQUIPMENT**

A designated area will be established to conduct cleaning of sampling and drilling equipment in the field prior to and between sample collection and between drilling locations. Prior to beginning any boring, all the drilling equipment that contacts the soil, rock or groundwater shall be decontaminated by pressurized steam-cleaning. This will include, but not be limited to, the drill casing, drill rods, drilling tools, and retrieval equipment. This operation shall be observed and approved by the inspecting geologist before drilling at each location proceeds.

## **DISPOSAL METHODS**

Rinse water, drill cuttings, PPE, and other residuals generated during the monitoring well installation will be managed in accordance with the materials management SOP.

Site Address: \_\_\_\_\_ Date: \_\_\_\_\_

Drilling Company: \_\_\_\_\_

Drilling Method: \_\_\_\_\_ Sample Method/Interval: \_\_\_\_\_ Boring Location \_\_\_\_\_

Depth (feet bgs)	Sample ID	PID Reading	Well Detail	LITHOLOGIC DESCRIPTION

Riser Materials: \_\_\_\_\_ Borehole Diameter: \_\_\_\_\_

Screen Material/Slot Size/Diameter: \_\_\_\_\_ Sand/Seal Types: \_\_\_\_\_

## **STANDARD OPERATING PROCEDURE FOR GROUNDWATER SAMPLING**

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### **PURPOSE AND SCOPE**

The purpose of this Standard Operating Procedure (SOP) is to provide information on the collection of groundwater samples. Low flow groundwater sampling should be done on the conventional monitoring wells if possible. Low flow groundwater sampling aims to collect groundwater samples that are “representative” of groundwater in the vicinity of the open well interval, at near natural flow conditions. In order to collect samples that are representative of the groundwater quality at the water table, the pump intake shall be located within the well screen, approximately two feet below the water level in the well and at least one foot below the top of the well screen. The minimum stress procedure emphasizes negligible water level drawdown and low pumping rates in order to collect samples with minimal alterations in water chemistry. The procedure is flexible for various well construction and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than five NTU and to achieve a water level drawdown of less than 0.3 feet during purging and sampling. If these goals cannot be achieved, sample collection can take place provided the remaining criteria in this procedure are met or, in cases where the well yield is insufficient to collect a sample using the low-flow sampling method, a bailer will be used.

### **EQUIPMENT**

- Adjustable rate, submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon), Peristaltic pump or Waterra pump;
- Tubing - Teflon or Teflon-lined polyethylene, PVC, Tygon, polyethylene (HDPE), or stainless steel tubing may be used;
- Power source (generator, compressed gas tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples;
- Water level measuring device - 0.01 foot accuracy. (Electronic devices are preferred for tracking water level drawdown during all pumping operations.);
- Indicator parameter monitoring instruments - pH (EPA Methods 150.1 or 9040), turbidity (EPA Method 180.1), specific conductance (EPA Methods 120.1 or 9050), and temperature (EPA Method 170.1);
- Optional Indicators - Eh and dissolved oxygen (EPA Method 360.1);
- Flow-through cell for measurement of indicator parameters.
- Standards to perform field calibration of instruments.
- Flow measurement supplies (e.g., graduated cylinder and stop watch);
- Decontamination supplies;
- Logbook(s), and other forms (e.g. low flow groundwater sample collection records);
- Sample bottles;

- Sample preservation supplies (as required by the analytical methods);
- Sample tags or labels;
- Well construction data, location map, field data from last sampling event; and
- Standard Operating Procedure.

## **INITIAL PROCEDURES**

Prior to collecting the sample, the following steps will be performed:

- Check well for damage or evidence of tampering. Record pertinent observations;
- Lay out sheet of polyethylene for monitoring and sampling equipment;
- If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one; and
- For conventional wells, measure and record the depth to water (to 0.01 feet) in the well to be sampled before any purging begins. Care should be taken to minimize disturbance of any particulate attached to the sides or at the bottom of the well.

## **PURGING**

Measure and record the water level again just prior to placing the pump in the well or removing any existing tubing from the well.

Lower pump, safety cable, and tubing, slowly into the well so that the pump intake is located two feet below the water table and at least one foot below the top of the well screen. Collection of turbid-free water samples may be difficult if there are three feet or less of standing water in the well.

When starting the pump, slowly increase the pump speed until discharge begins. Check water level. Adjust pump speed until there is little or no water level drawdown. The target drawdown is 0.3 feet and it should stabilize. If the target of less than 0.3 feet drawdown cannot be achieved or maintained, the sampling is acceptable if remaining criteria in the procedure are met as long as the drawdown does not exceed two feet. If drawdown reaches the pump intake follow the bailer sampling protocol described below.

Monitor water level and pumping rate every three to five minutes (or as appropriate) during purging. Record pumping rate adjustments and depths to water. Pumping rates should be reduced, as needed, to the minimum capabilities of the pump (e.g., 0.1-0.2 gal/min.) to ensure stabilization of indicator parameters. Adjustments are best made in the first 15 minutes of pumping in order to help minimize purging time. During initial pump start-up, drawdown may exceed the 0.3 feet target and then recover as pump flow adjustments are made (minimum purge volume calculations should utilize stabilized drawdown values, not the initial drawdown). If the recharge rate of the well is less than the minimum capability of the pump do not allow the water level to fall to the intake level. If the water level is drawn down to the pump intake, shut off the pump and allow the water level to recover. Repeat the process until field indicator parameters stabilize and the minimum purge volume is removed. The minimum purge volume with negligible drawdown (0.3 feet or less) is the volume of the drawdown plus the volume of the



tubing. After the minimum purge volume is attained and field parameters have stabilized, begin sampling. For low yield wells, commence sampling as soon as the well has recovered sufficiently to collect the appropriate volume for all anticipated samples.

In cases where the well yield is insufficient to collect a low-flow sample allow the water level to recover over night and collect the sample using a bottom discharge bailer. The bottom of the bailer will be lowered two feet below the water table.

## **FIELD PARAMETER STABILIZATION AND SAMPLE COLLECTION**

During well purging, monitor field indicator parameters (turbidity, temperature, specific conductance, pH, etc.) every three to five minutes (or as appropriate). Purging is complete and sampling may begin when all field indicator parameters have stabilized as follows for three consecutive readings taken at three- to five-minute intervals:

- turbidity: within 10% for values >1 NTU
- DO: within 10%
- specific conductance: within 3%
- temperature: within 3%
- pH:  $\pm 0.1$  unit
- Eh:  $\pm 10$  millivolts

If the parameters have stabilized, but turbidity remains above the five NTU goal, decrease pump flow rate, and continue measurement of parameters every three to five minutes. If the pumping rate cannot be decreased any further and stabilized turbidity values remain above the five NTU goal, record this information and collect the samples. If the parameters have not stabilized after three screen volumes have been removed or four hours of purging have occurred, record this information and collect the samples. Measurements of the field parameters should be obtained using a flow-through cell. Prior to collecting the samples for laboratory analyses, the flow-through cell must be disconnected. Note, turbidity, temperature, specific conductance, and pH measurements must be recorded. If these measurements are missing, the resulting sampling data may not be acceptable

Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

1. Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel), or clamp which should reduce the flow rate by constricting the end of the tubing;
2. Insert a narrow diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing; collect sample from the narrow diameter tubing.

Collect samples in pre-preserved vials provided by a certified analytical laboratory.

Label each sample as collected. Samples requiring cooling will be placed into an ice cooler for delivery to the laboratory.

After collection of the samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth.

Secure the well.

## **FIELD INSTRUMENTATION CALIBRATION**

There is a wide variety of meters and other equipment that is used in making field parameter measurements. pH, Eh, specific conductance, dissolved oxygen, turbidity and temperature are collected for each monitoring well sampled. Field parameters aid in determining adequate well purging prior to sampling. The stability of these parameters while purging is the usual criteria for this determination. These parameters are also gross indicators of water quality. All monitoring instrumentation must be operated in accordance with USEPA methods and the operating instructions provided by the manufacturer.

Most meters provide a method of calibration against a standard so that accurate readings can be taken. Temperature is usually measured with a meter that measures another parameter and usually cannot be calibrated. Temperature probes should therefore be checked against an NIST traceable thermometer to account for any inaccuracies of the field probe.

The field parameters, pH, Eh, specific conductance, dissolved oxygen, turbidity and temperature will be measured at each groundwater sampling location according to the following procedures:

### **pH, Eh, Specific Conductivity and Temperature Meter**

- Prepare and standardize the meter(s) for use according to the manufacturer's instructions and EPA methods (Use pH standard buffer solutions with pH of 4, 7, 10, Eh standard with redox potential of  $231 \pm 10$  mV and specific conductivity solution with specific conductance of 70, 445, 2060, 7000 micromhos);
- Rinse the probes with distilled water and blot dry;
- Insert probes into flow-through cell;

### **Dissolved Oxygen Meter**

- Prepare the dissolved oxygen probe for use according to the manufacturer's instructions taking special precaution that the membrane is in good condition;

- Prepare the instrument for operation according to the manufacturer's instructions. Before beginning calibration leave the meter on for at least 15-minutes to allow the probe to stabilize;
- Calibrate the meter using the manufacturer's instructions for air calibration;
- Insert the probe into the flow-through cell.

### **Turbidity Meter**

- Prepare and calibrate the meter according to the manufacturer's instructions and USEPA Method 180.1 using a 1 NTU standard;
- Confirm the calibration with a 10 NTU standard;
- At all times during calibration and operation make sure that the cuvettes are not scratched and that the outside is dry, free of fingerprints and dust;
- Before making a turbidity measurement rinse the cuvette with the sample;
- Following the measurement rinse the cuvette with deionized water.

All field meters should be calibrated at the beginning of the day and confirmed at the end of each day. Calibration of field meters is to be documented in the field note book. Field meters should be kept clean and dry and be protected from damage at all times.

### **DECONTAMINATION**

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. Pumps will not be removed between purging and sampling operations. The pump and tubing (including support cable and electrical wires which are in contact with the well) will be decontaminated by one of the procedures listed below.

#### **Procedure 1**

- Steam clean the outside of the submersible pump.
- Pump hot water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three- or four-inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. Hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.
- Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.
- Pump tap water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.
- Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

## **Procedure 2**

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and isopropanol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is flushed out. The outside of the pump and the electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

- Flush the equipment/pump with potable water;
- Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.;
- Flush with tap or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.;
- Flush with isopropanol. If equipment blank data from previous sampling show that the level of contaminants is insignificant, then this step may be skipped;
- Flush with distilled/deionized water. The final water rinse must not be recycled.

## **FIELD QUALITY CONTROL**

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation.

Sampling should proceed from wells with the lowest contaminant concentration to the highest concentration. Collect equipment blanks after sampling contaminated wells and not after sampling background wells. When field duplicate or split samples are to be collected, they will be collected consecutively for the same parameters.

All monitoring instrumentation must be operated in accordance with USEPA analytical methods and the operating instructions as supplied by the manufacturer. The instruments must be calibrated at the beginning of each day and the calibration checked at least once throughout the day (i.e. at the end of the day) to verify that the instruments remain in calibration. Temperature measuring equipment, thermometers and thermistors, should be checked for accuracy prior to field use according to the USEPA Method 170.1 and the manufacturer's instructions.

## **FIELD LOG**

A field log must be kept each time groundwater monitoring activities are conducted in the field. The field log should document the following:

- Well identification;
- Well depth and measurement technique;

**Title:** QAPP for Vapor Intrusion Assessment

**Site Name:** Wells G&H Superfund Site

**Site Location:** Woburn, Massachusetts

**SOP 3**

**Revision Date:** 3/25/10

**Revision Number:** 0

- Pre-pumping water level depth, date, time, and measurement technique;
- Pumping rate, drawdown, indicator parameter values, and clock time at the appropriate time intervals; calculated or measured total volume pumped
- Well sampling sequence and time of sample collection;
- Types of sample bottles used and sample identification numbers;
- Preservatives used;
- Parameters requested for analysis;
- Field observations of sampling event;
- Name(s) of sample collector(s);
- Weather conditions; and
- QA/QC data for field instruments.

## **DISPOSAL METHODS**

Rinse water, purge water, PPE, and other residuals generated during the equipment cleaning will be managed in accordance with the Materials Management SOP.

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**GeoTrans, Inc.**

**Title:** QAPP for Vapor Intrusion Assessment  
**Site Name:** Wells G&H Superfund Site  
**Site Location:** Woburn, Massachusetts

**SOP 4**  
**Revision Date:** 3/25/10  
**Revision Number:** 0

## **STANDARD OPERATING PROCEDURE FOR MATERIALS MANAGEMENT**

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The investigation derived waste (IDW) generated during the VIA will be managed in a manner consistent with Resource Conservation and Recovery Act (RCRA) and Superfund Amendments and Re-authorization Act (SARA). All IDW generated during VIA activities at the Site will be properly managed. We anticipate that the following types of waste will be generated: (1) non-hazardous solid waste, such as packaging and paper waste, (2) non-hazardous and hazardous personal protective equipment and disposable sampling equipment, (3) decontamination fluids, (4) purge and development waters from the monitoring wells, and (5) drill cuttings from well drilling.

Non-hazardous solid waste, personal protective equipment and disposable sampling equipment will be collected in plastic bag-lined garbage cans, removed from the Site, and regularly disposed of at a non-hazardous waste landfill or incinerator. Personal protective equipment and disposable sampling equipment will be deemed non-hazardous based on the use of the equipment and, if necessary, laboratory analysis.

Decontamination fluids containing isopropanol will be drummed and stored near the Grace treatment building. Other decontamination fluids and purge and development water will be contained and treated at the Grace or UniFirst groundwater treatment system.

Disposal of cuttings from wells installed during the VIA will be determined based on headspace screening. Drill cuttings with a PID headspace reading less than 10 ppmv will be spread on the ground on the Grace property. Drill cuttings with a headspace reading greater than 10 ppmv will be drummed and stored near the Grace treatment building. The drums will be labeled with the well identification number and the dates of accumulation. At the completion of the drilling, a representative sample of cuttings from each drum will be analyzed and the drums will be disposed of in an appropriate manner.